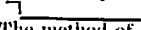
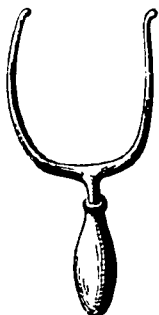


I am also having a glass hood condenser made which will fit into a depression in the cover, the cover being made so  to receive it.

The method of using the apparatus is this:—Two of the basins containing a sufficient quantity respectively of weak and strong nitric acid are heated over two of Fletcher's radial burners, the basin of weak acid being covered with the perforated plate carrying the tubes, and the strong acid being covered with an ordinary dinner plate. The assays, which are flattened, &c. whilst the acid is heating, are dropped into the tubes, and as soon as the action of the acid has ceased the cover is lifted off by means of a metal crutch covered with india-rubber, and the tubes are dipped

Fig. 3.



into a basin of distilled water. The whole 30 being thus washed at one operation, they are then transferred to the basin of strong acid (sp. gr. 1.28) and are boiled briskly for half an hour, the time being noted on a small slate hung at the side of the draught closet.

It is very necessary that the boiling should be brisk, as otherwise a much greater surcharge will be found, and it is curious to note that this was known at the time Erekers wrote, for he says, "Take notice also that you may cause the last aqua fort to work in great bubbles that the roles may become clean."

After boiling in the second acid, the tubes are lifted by the cover and are dipped into distilled water and allowed to drain. Each tube is then taken out in order and is plunged over head in a basin of distilled water, so as to completely fill the tube. The tube is then inserted over a small porous crucible and the assay allowed to fall through the water into the crucible. The crucibles are drained and are then annealed, *i.e.*, ignited in the muffle, when the assays are ready to weigh. The use of somewhat similar perforated tubes had been tried for this purpose some time ago, but was given up it is said from the difficulty which was found in transferring the assay to the crucible. In the method described above there is no difficulty whatever in doing this, and when the writer designed this apparatus he had no knowledge that tubes of this kind had been used before.

This apparatus has now been in use at the Chester Assay Office for the last 10 years, during which time at least 90,000 assays have been made with it, and has given every satisfaction. Its great advantages are that all the assays are under precisely the same conditions, it is a very great saving of labour and time, much less nitric acid is required, and it requires no attention until it is time to take off the assays; in fact it combines nearly all the advantages of the costly platinum cups and boilers used for a similar purpose with a very moderate cost.

The only special precautions to be taken with it are: (1) to have the first acid sufficiently dilute; (2) to see that the second acid boils properly; and (3) not to boil the second acid longer than about half an hour, as the best results are obtained under these conditions.

Pure gold check assays are employed with each batch of assays, for in putting gold and silver with nitric acid there is always a slight surcharge with the higher standards and a slight loss with the lower, and this has to be allowed for in weighing up the finished assays. In the process described

the surcharge on 22 carat or $\frac{916.6}{1,000}$ gold is usually from 0.06 to 0.07 per 1,000. On 15 carat or $\frac{625}{1,000}$ gold and on 18 carat or $\frac{750}{1,000}$ gold it is usually nil, but if the acid has not been boiling well, there will be a surcharge. With 9 carat or $\frac{325}{1,000}$ gold there is a slight loss, usually about 0.06 to 0.08 per 1,000. Under the proper conditions both surcharge and loss are very regular. The surcharge is due to the retention of silver in the cornets, and this was known as far back as the time of Erekers, for he recommends the use of check assays.

The pure gold used in check assays is prepared by dissolving fine gold (assay bits) in aqua regia, evaporating off the acid, and diluting very largely with water to allow all the silver chloride to settle. The clear solution is then siphoned off, and the gold precipitated with oxalic acid. The precipitated gold is boiled with hydrochloric acid, to free it from any traces of silver chloride. It is then washed with hot water, dried, and fused under bisulphate of potash.

In preparing some pure gold in this way from 5 oz. of assay bits, 3.858 grs. of AgCl were obtained. This is equal to 2.9 grs. of silver; so that the amount of silver present in the assay bits was equal to 1.23 parts per thousand.

In cupelling gold there is always a slight loss, due to absorption into the cupel. It is usually stated that this loss is greater with the lower than with the higher standards. This, however, does not appear to be the case, for the following table give the results of some assays made of cupels which had been used for different standards, and it will be seen that the absorption is almost identical.

Standard.	Weight of Gold obtained from 1 Cupel (= 4 Assays of 5 grs. each).	Absorption per Thousand.
9 carat, or $\frac{375}{1,000}$	0.014 grs.	0.7
Do. do.	0.014 "	0.7
15 carat, or $\frac{625}{1,000}$	0.014 "	0.7
Do. do.	0.015 "	0.75
18 carat, or $\frac{750}{1,000}$	0.014 "	0.7
Do. do.	0.015 "	0.75
22 carat, or $\frac{916.6}{1,000}$	0.014 "	0.7

The above were all cupelled on different days, so that the temperature of the furnace would not account for this regularity; but as the amount of lead used is the same for all standards, this, most probably, is the cause of the regularity.

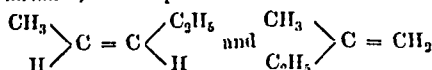
The number of assays made last year at the Chester Assay Office, from articles sent to be hall-marked, was 25,100, of which more than half were gold.

PROPOSED UNIFICATION OF NOMENCLATURE IN ORGANIC CHEMISTRY. (PARIS.)

The following is the text of the resolutions adopted at the International Chemical Congress recently held in Paris (this Journal, 1889, 656).

Nomenclature of the Non-saturated Derivatives of the Fatty Series.—"The letters *a* and *b* shall designate the two atoms of carbon bound together by multiple links, in the case of bi-substituted derivatives; in the case of tri-substituted derivatives the designation may be omitted, provided the two radicals united to the same atom of carbon be named first."

For instance, the compounds—



shall henceforth be known as (1) α -methyl-*b*-ethyl-ethylen,
(2) α -methyl-ethyl-ethylen.

The same rule shall apply to bi-substituted ureas.

Nomenclature of Aldehydes.—"Aldehydes shall henceforth bear the name of the corresponding alcohols, instead of the acids." Hence we shall have methylic, ethylic, and benzylic aldehydes, instead of formic, acetic, and benzoic aldehydes. A good reason for the change is that the word aldehyde is only an abbreviation for de-hydrogenated alcohol.

Nomenclature of Acetones.—"In the designation of acetones, for the word carbonyl, which is liable to misunderstanding, the term cetone shall be substituted, as it has no special meaning as yet, and corresponds to the word *ketone* used in German literature."

Consequently ordinary acetone, $\text{CH}_3\text{—CO—CH}_3$, will be called dimethylketone, instead of dimethylcarbonyl. In the same way the compound $\text{CH}_3\text{—CH}_2\text{—CO—CH}_2\text{—CH}_3$, hitherto indiscriminately called dimethylacetone and diethylcarbonyl, will definitely be known as diethylketone.

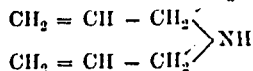
Designation of the group CN.—"The group CN shall keep the name of cyanogen when substituted to an atom of hydrogen." Hence, for instance, the compound C_2H_5-CN , known as ethyl cyanide or propionitrile indifferently, will henceforth bear the name of cyanethan.

Value of the termination ol.—"In the designation of hydrocarbons the termination *ol* shall be entirely discarded, and exclusively reserved for alcohols and phenols. The names of aromatic hydrocarbons shall always have the desinenae *en*." [Note.—In the French text the desinenae is spelled *ene*, which in Latin languages has for equivalent *ena* or *eno*, and in the Germanic *en*. The last form has in this article been preferred to *ene*, which in English, owing to the absence of accents, is liable to be mistaken for *ine*.]

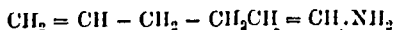
According to the foregoing rule, durol and naphthuline will become duren and naphthalen, and so on with others.

Firing the value of the prefixes bi and di.—"When designating bodies formed by a duplication of the radicals, the prefix *bi* shall henceforth be employed, and *di* be kept for double substitutions; according to this convention some names already adopted are to be gradually transformed, and diphenyl, dipropagyl, &c., for instance, shall be known as biphenyl, bipropagyl, &c."

One example will suffice to illustrate this important resolution. Let us take the two following compounds:—(1)

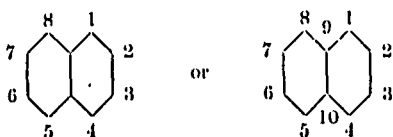


and (2)

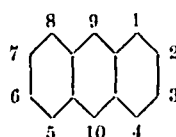


each containing allylic groups. The first, being an amine twice substituted by the allyl radical, will be called diallylamine. The second, being an amine substituted by one group resulting from a duplication of the allyl radical, will be named bi-allylamine.

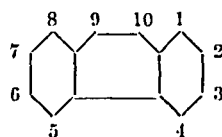
Nomenclature of complicated Nuclei like Naphthalen, Anthracen, &c.; Designation of the Apexes.—Complicated nuclei are, it is known, represented by regular geometric figures. When compounds derived therefrom are to be named, it is necessary to indicate on which apex the substitution of hydrogen by a radical has taken place. Such points may be designated either by numbers or by Roman or Greek letters. The congress has decided in favour of Arab numbers, and adopted the following order for the principal nuclei:—



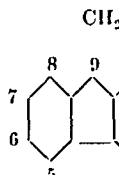
For naphthalen (or naphthalino).



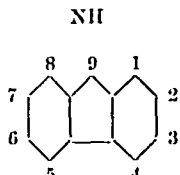
Anthracen,



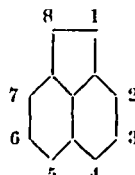
Phenanthren.



Fluorene.



Carbazol.



Acenaphthen.

The programme bore also other desiderata, such as a system of notation for nitro-compounds and for nuclei containing other atoms than carbon and hydrogen, but the questions were not considered mature enough for a final decision, and were consequently referred to some future international congress or commission. It is hoped also by pharmacists that chemists, leaving aside for a moment pure theory, might think of practical concerns, and take initial steps towards a simplification of nomenclature, so that currently-used chemicals should have names not exceeding four or five syllables. But the programme was already so heavy that it became impossible to consider outside questions, and the much desired improvement was left in abeyance. It is, perhaps, unjust to ask or expect too much of one congress. So let this be praised for the good work it has done. If the resolutions adopted be generally accepted, as it is hoped they will be, important progress will have been secured towards uniformity in organic nomenclature, and simplicity will afterwards be more easily obtainable.

Journal and Patent* Literature.

I.—GENERAL PLANT, APPARATUS, AND MACHINERY.

The Design and Construction of Chimney Shafts. Industries, 7, 224.

(See pages 692—693.)

PATENTS.

Method of Producing Solid Steel Castings and Apparatus therefor. B. H. Thwaite, Liverpool. Eng. Pat. 8095, June 4, 1888. 11d.

This patent relates to the production of solid steel castings free from internal cavities by means of moulds with special flues, &c., so arranged that the metal when poured is subjected to a high temperature on the one side and to a chilling influence on the other. Sixteen drawings are given in the specification, showing the different arrangements by which this is effected.—A. W.

* Any of these specifications may be obtained by post, by remitting the cost price, *plus* postage, to Mr. H. Rendor Lack, Comptroller of the Patent Office, Southampton Buildings, Chancery Lane, London, W.C. The amount of postage may be calculated as follows:—

If the price does not exceed 8d.	1d.
Above 8d., and not exceeding 1s. 0d.	1d.
" 1s. 0d., " " 2s. 4d.	11d.
" 2s. 4d., " " 3s. 4d.	2d.

The Design and Construction of Chimney Shafts.

EXAMPLES OF EXISTING

For whom Constructed.	External Form of Shaft.	Total Height, including Foundations.	Height from Ground Line.	Outside Dimensions of Foundations.	Outside Dimensions of Shaft at Ground Line.	Outside Dimension of Shaft at Top.	Inside Dimension of Flue at Ground Line.	Inside Dimension of Flue at Top.
Mr. Townsend, Chemical Works, Port Dundas, Glasgow. <i>a</i>	Circular (brick).	468'	451'	50' (circular).	32'	13' 4"	17' 10"	11'
Messrs. Tennant and Co., St. Rollox, Glasgow. <i>b</i>	Circular (brick).	455' 6"	435' 6"	50' (circular).	40' diameter.	13' 6" diameter.	12'	11' 2"
Mechernich Lead Mining Co., near Cologne, Rhenish Prussia. <i>c</i>	Circular stalk on square pedestal, 32' 8" high (brick).	441' 48'	430'	39' 30" (square).	32' 8" (square).	11' 48'	11' 45'	9' 8 1/4'
Messrs. Johnson & Co., Cement Works, Greenhithe, Kent. <i>d</i>	Circular (brick).	304'	297'	30' (square).	25'	11'	17' 6"	8' 9"
Dowlais Iron Works, South Wales. <i>e</i>	Circular stalk on square pedestal, 48' high (brick).	284' 5"	268' 8'	42' (square).	32' (square).	17' 8"	19'	15' 4"
Amoskeag Manufacturing Co., Manchester, New Hampshire, U.S.A. <i>f</i>	Circular (brick).	265'	255'	25' 8"	25'	12' 6"	16' 8"	10'
West Cumberland Hematite Iron Works. <i>g</i>	Circular (brick).	267'	250'	34' 6" (square).	25' 7"	15' 3"	21' 10"	13'
Deptford Pumping Station, Metropolitan Main Drainage. <i>h</i>	Circular stalk on square pedestal, 36' high (brick).	160' to top of concrete bed.	150'	22' (square).	18' (square).	8' 3" diameter.	7' 6"	6'

EXAMPLES OF EXISTING

*Messrs. Cox Brothers, Camperdown Linen Works, Lochec, Dundee. <i>i</i>	Ornamental (brick), square for 230', remainder octagonal.	296'	282'	35' (square).	30'	19' (octagonal).	14' 6" diameter.	13' 8" diameter.
*Messrs. Lister & Co., Manningham Mills, Bradford. <i>j</i>	Square ornamental (stone).	262' 6"	256' 6"	40' (square).	21'	21'	10'	13'
Crossness Pumping Station, Metropolitan Main Drainage. <i>k</i>	Ornamental (brick) stalk, with curve base on square pedestal.	227' to top of iron cap, 201' to bottom of iron cap.	162'	30' 2" by 25' 6"	26' 6"	12' below cap.	8' 5" diameter.	8' 3" diameter.
Western Pumping Station, Metropolitan Main Drainage. <i>l</i>	Square (brick), no pedestal, ornamental cast-iron cap.	201' 3"	172' 9"	35' (square).	26' 9"	15' 2"	7' 3'	7'
Kent Brick and Tile Company, Pluckley. <i>m</i>	Square (brick).	..	170'	..	10' 6"	5' 6"	7' 6"	4' 6"
Messrs. Stansfield and Co., Wallham Green, London. <i>n</i>	Ornamental design, square stalk on pedestal 32' 6" high (brick).	152'	120'	25' (square).	15' 6"	5' 6"	4' (square).	4' (square).

EXAMPLES OF EXISTING

*Messrs. Wessensfield's Chemical Factory, Harmon, Prussia. <i>a</i>	Octagonal stalk on square pedestal, 40' high (brick).	315'	331'	..	20'	11'	8'	8'
*Messrs. Crossley and Sons, Dean Clough Mills, Halifax. <i>c</i>	Octagonal (stone).	330'	300'	32'	30'	15' 6"	9'	9'
*Messrs. Storey Brothers, White Cross-street Mills, Lancaster. <i>p</i>	Octagonal (brick) stalk, with stone cap.	270'	250'	28' (square).	25'	10' 8"	8'	8' (octagonal); top 1' 2", bottom 2"
Abbey Mills Pumping Station, Metropolitan Main Drainage. <i>q</i>	Octagonal stalk on square (stone) pedestal, 30' high.	212'	101'. Ground line to top of stone cap, 158' 3".	37' 6" (square).	36' 6"	10' 3" (underside of stone cap).	5'	8'

a Designed by Professor Rankine and R. Corbett. Largest shaft existing.*c* Designed by Mr. Martin.*f* Designed by Messrs. Stevens & Manning.*g* Designed by Professor Rankine.*h* Designed by Sir J. Bazalgette. Brickwork in foundation and pedestal is built cellular.*m* Designed by Mr. Ward. Peculiarity of this shaft is that it is built of 4 1/2 in. cellular brickwork.*p* Designed by Mr. Storey and Messrs. Paley and Austin.*q* Designed by Sir J. Bazalgette. Chief feature was

C. Molyneux and J. M. Wood. Industries, 7, 224.

ROUND CHIMNEY SHAFTS.

Ratio of Height to Width externally, immediately above the Footings.	Batter of Shaft.	Weight of Shaft.	Cost.	Maximum Pressure per Sq. Ft. on Brick-work and Foundations.	Thickness of Brick-work, and Number of Sections into which the Shaft is Divided.	Lining (internal).	Duty.
14 to 1	1 in 48½	8,000 tons.	8,000l.	4.07 tons on foundations.	12 sections, 5' 7" thick at bottom, 1' 2" at top.	9" fire brick with 9" air space, 60' in height.	Gases from chemical works.
11 to 1	1 in 32.9	4,100 tons (about).	..	3 tons on top of concrete foundations.	5 sections, 2' 7½" thick at bottom, 1' 2" at top.	Inner lining, 263' high, 1' 10½" at base, 1' 2" at top.	..
13.1 to 1	1 in 60½ (circular stalk).	5,459 tons.	..	Base of stalk 0.191 tons, 3.7 tons on foundations.	27 sections, 11.48' thick in pedestal, 6' 56" at base, 0' 82" at top.	No lining.	Fumes from lead works.
11.9 to 1	1 in 41	2,500 tons (about).	2,500l. (about).	1½ tons (about).	8 sections, 3' 9" thick at bottom, to 1' 2" at top.	No lining.	Steam and fumes from cement kilns.
8.5 to 1	1 in 38½ (circular stalk).	..	..	..	8 sections, 7' thick in pedestal, 4' 6" at base of stalk, 1' 2" at top.	No lining.	Waste gases from boilers, &c.
10.2 to 1	1 in 40.8	2,330 tons.	3,333l.	4.5 tons on foundation.	2' 5" thick at bottom, 1' 3" at top.	Lining, 10' diameter throughout.	Fumes from 64 boilers=8,400 h.p. Designed to burn 18,000 lb. of anthracite per hour.
8.75 to 1	1 in 49	2,380 tons (about).	1,500l. or 4d. per cubic foot of the whole space occupied by the building.	At springing at flue arches 3 tons, 1.6 tons on foundation.	4 sections, 3' thick at bottom, 1' 10½" at ground line, 1' 2" at top.	Fire brick lining top to bottom. Upper 160' ½ brick thick, rest 1 brick.	Gases from four blast furnaces, stoves, and boilers. 10½ tons of fuel per hour.
8.6 to 1	1 in 56½ (circular stalk).	..	..	..	4 sections, 5' 3" thick at ground line, 1' 10½" at base of stalk.	4½" fire brick lining carried up 36" above ground line.	10 Cornish boilers, 30' long by 6' diam.

SQUARE CHIMNEY SHAFTS.

9.5 to 1	Vertical sides with external offsets.	..	..	..	12' thick at foundation, 8' at ground line, 3' 5" 47' from ground line, 1' 6" at top.	Inner flue, circular from top to bottom, 1' 6" thick at bottom, 0' at top.	58 furnaces, smiths' forges, &c.
12.2 to 1	Vertical sides.	8,000 tons (about).	10,000l. (about).	5 tons (about) on foundation.	..	6" fire brick, 50' in height, 4" air cavity.	..
7.8 to 1	Curved batter for 42' high, remainder of stalk vertical.	..	4,000l. (about).	..	5' thick at base of stalk, 1' 10½" where curve ends, this thickness carried up throughout.	Fire brick lining, carried up 40'.	12 Cornish boilers, 6' diam. by 30' long.
8.4 to 1	1 in 62½	..	..	..	3' 5½" thick at ground line, 1' 2" at top.	Inner flue 14" thick from bottom to top, 4½" fire brick for 40'.	18 Lancashire boilers, 22' long by 6' 6" diam.
10.3 to 1	1 in 31	..	..	..	4½" thick throughout.	..	Gases from Hoffmann's brick kilns.
8 to 1	1 in 70 (square stalk).	..	1,000l. (about).	..	5 sections, 5' 7" thick in pedestal, 1' 10½" at base of stalk, 0" at top.	4½" fire brick lining, 33' above ground line.	2 Lancashire boilers, 2 brewing coppers.

OCTAGONAL CHIMNEY SHAFTS.

10.5 to 1	1 in 97	..	..	9½ tons on lowest part of chimney.	5' 3" thick in pedestal, 3' 9" at base of octagon, 1' 6" at top.	..	Products from chemical factory.
10 to 1	1 in 41	8,300 tons.	9,000l. to 10,000l.	8 tons (about) on foundation.	..	Inner circular shaft of 14" fire brick, with a 3" air space.	Products from 15 boilers, &c.
19 to 1	1 in 54 (about).	3,300 tons.	2,800l.	4½ tons (about) on foundation.	Outer shaft, 4' 4" at bottom, 1' 2" 20' from top, 9" at top.	Inner shaft 26½' high, 18" thick at base, 9" at top, built parallel.	Products from boilers.
..	1 in 100 for a height of 78' below cap.	Stone cap, 60 tons; iron cap, 27 tons.	Brickwork in stalk cost about 8l. per rod (labour only).	..	3 sections: (1) First 40', 4' 4½" to 1' 10½"; (2) 1' 4½"; (3) 1' 2" thick.	..	6 Lancashire boilers, 8' diam. 40' long.

and Hills. Second tallest shaft existing.

A Designed by Sir J. Bazalgette.

I Designed by Sir J. Bazalgette.

N Designed by Mr. Bradford.

cast iron cap, which has since been removed owing to chimney being much cracked.

a Third tallest shaft existing.

I Designed by Mr. Macdonald.

o Designed by Messrs. R. Ives & Sons.

d Designed by Mr. Johnson.

j Designed by Messrs. Andrews & Pepper.

Chief feature of this shaft is a stone spiral staircase, built in the space between the lining and outer shell.

* Authority, Hancock.

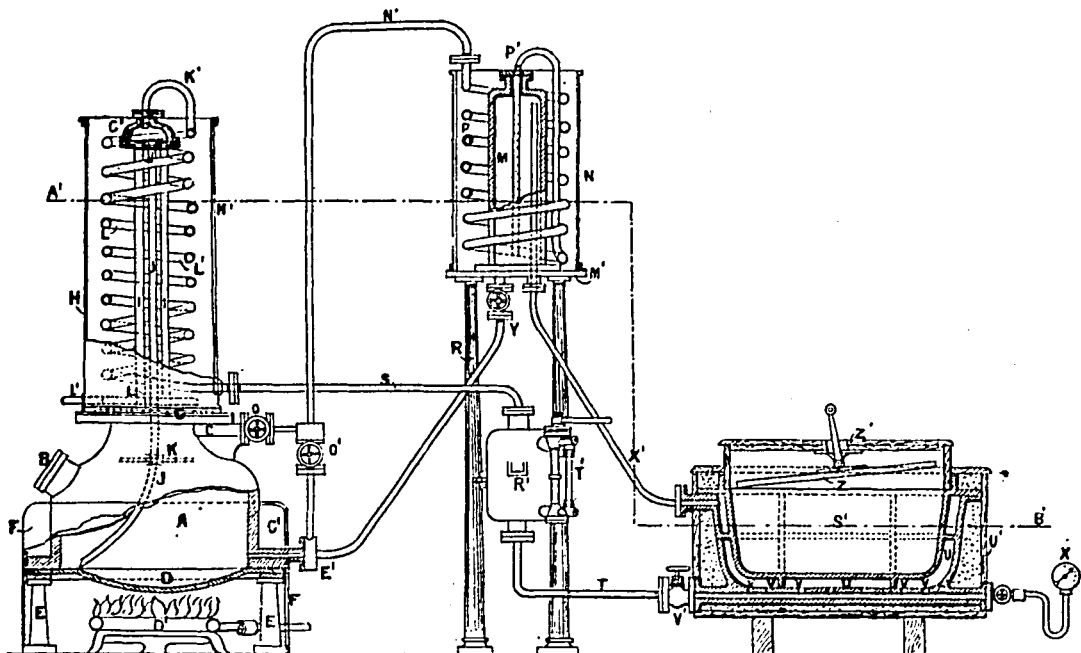
Improvements in Filters. M. Negro, London. Eng. Pat. 11,357, August 7, 1888. 8d.

Two annular tubes of perforated metal or wire-work enclose between them the filtering agent, which may be paper, asbestos, or other of the substances commonly used for such a purpose. The filter so formed is fitted by means of a screwed socket in the interior of a conical vessel forming the reservoir for the unfiltered substance, the lower end of which forms the outlet for the filtrate.

—C. C. H.

An Improved Apparatus for Ice-making and Refrigerating. A. Glasson, London. Eng. Pat. 12,008, August 20, 1888. 8d.

THE improved refrigerator belongs to the type generally known as "ammonia absorption" apparatus. A is the generator, heated preferably by gas, and vaporising the concentrated solution of ammonia it contains. The vapour rises through the rectifier tubes I into the bonnet G'; the water condensed returns through pipe J, and the vapour passes on, is condensed in worm L, and, by means of tube S, delivered into receiver R' supplying the freezing cell S'.



Vaporising again in jacket U, it abstracts heat from the water contained in S', kept in motion by stirrer Z, and, passing through pipe X' into absorber M, meets with a fine spray of weak liquor delivered from A through tube N'; cooled in worm P, is absorbed and again let down into A when required to be re-distilled.—C. C. H.

Improved Method of and Apparatus for Centrifugally separating Solids and other Matters from Liquids. W. Huicks, London. Eng. Pat. 2528, February 13, 1888. 8d.

A NUMBER of arms or chambers radiating from a common centre, provided internally with partitions so as to form a zigzag passage from the upper to lower part, are fixed to a vertically revolving shaft. The outer extremities are fitted with sliding valves, so that the ends of the horizontal chambers formed by the partitions can be opened; the lower ends of all the chambers formed by the partitions communicate with a common overflow pipe next the rotating shaft. The action is as follows:—Liquid containing solid matter is introduced into the upper part of the chambers, the solid matter it contains by centrifugal force collects on the periphery and may be discharged from time to time through the sliding valves, whilst the liquid passes to and fro from centre to periphery and *vice versa*, and so on through the entire length of the passage in the chambers, and finally overflows in a clarified condition into the common outlet pipe. A modification is also shown, in which the liquid is forced in, and so urged filtration or separation secured.—C. C. H.

II.—FUEL, GAS, AND LIGHT.

Experiments on the Manufacture of Oxygen in Gasworks, and the Result of its practical Application to the Purification of Coal Gas. W. A. Valon. The Gas World, 1889, 715—719.

IN a previous paper the author has given an account of experiments in the purification of gas by means of oxygen in conjunction with "oxide" or lime. When oxide alone is employed and a small quantity of oxygen is mixed with the crude gas, the oxide is revived *in situ*, the life of the purifier is lengthened, and the cost of labour is considerably diminished. The advantages of using oxygen are still more marked when lime, in quantity sufficient to remove the carbon dioxide, is employed in purification; not only the carbon dioxide but the whole of the sulphur (sulphuretted hydrogen) and some of the sulphur compounds are removed, the latter being abstracted with perfect certainty, and the lime having no unpleasant odour either when partially or completely spent.

The oxygen is made by Brin's process, and the necessary apparatus is fully described. The barium oxide is heated at a constant temperature in steel retorts placed in ordinary gas retorts, and by the aid of an automatic arrangement for reversing the valves, devised by Mr. Murray, the process may be described as practically automatic. The requisite quantity of oxygen is mixed with the gas by means of a proportional meter, devised by W. G. Hicks, which automatically and accurately regulates the percentage of oxygen necessary for purification, and is so arranged that the quantity added can be increased or decreased at will.

In experiments made at the Ramsgate works, a series of four purifiers (1, 2, 3, 4), 14 ft. x 14 ft., were each charged with 12 yds. of slacked chalk lime, not 60 per cent. of

which was caustic, and the gas, previously mixed with 0.6 per cent. of oxygen, passed through three of the boxes. The test for foulness is carbon dioxide in equal quantities at the inlet and outlet; with Sheard's apparatus a test can be made in a few minutes. The foul gas at the inlet of the first purifier contained on the average a trifle under 1 per cent. of carbon dioxide and 600 grains of sulphuretted hydrogen per 100 cubic feet. For the first six days the gas at the outlet of No. 1 was free from both sulphuretted hydrogen and carbon dioxide, and on the seventh day it contained only 98 grains of carbon dioxide and 140 grains of sulphuretted hydrogen; the inlet at the foul main showed at the same time 700 grains of carbon dioxide and 600 grains of sulphuretted hydrogen, and the sulphur (sulphur compounds) in the purified gas was under 6 grains. On the seventh day No. 2 commenced to work, and no carbon dioxide or sulphuretted hydrogen were present at the outlet until the 14th day. On the 14th day No. 3 commenced to work, and, 29 days after No. 1 was started, the gas at the outlet of No. 3 contained only 5 grains of sulphuretted hydrogen and not a measurable trace of carbon dioxide, No. 1 not yet being quite exhausted, although having passed more than 8 million cubic feet of gas. No. 1 was finally thrown out on the 40th day, 9 million cubic feet of gas having passed through it. The gas thus freed from sulphuretted hydrogen and carbon dioxide completely, and from sulphur compounds to a considerable extent, was, therefore, more than half a million cubic feet per yard of caustic lime, and the total sulphur in the purified gas did not at any time exceed 8 grains per 100 cubic feet.

These facts prove beyond doubt (1) the instantaneous action of oxygen; (2) the fixity of the sulphur in the lime; (3) that with oxygen a greatly reduced area is required; (4) economy in lime; (5) economy in labour; (6) simultaneous and perfect purification from sulphuretted hydrogen and carbon dioxide; (7) easy and absolute control of the sulphur compounds without trouble or anxiety; and (8) complete immunity from anything objectionable while carrying out the operation.

Experiments which were made to ascertain the effect of air on the illuminating power gave the following results:—The gas at Ramsgate has averaged for years past, with oxide purification and 1 per cent. of air, 13.5 candles, the coal used being Pelaw Main, and the make over 10,000 cubic feet per ton; 2½ per cent. of New Boghead or Tyne cannel are added to bring the illuminating power up to 15.5 candles. Since lime and oxygen have been employed the illuminating power has ranged between 16.5 and 17.3 candles, and no cannel whatever has been used, so that, allowing that the increase of one candle is due to the removal of 1 per cent. of carbon dioxide, the difference of 2½ candles may be credited to the use of 0.6 per cent. of oxygen and the discontinuance of 1 per cent. of air.

The effect of various quantities of air on the illuminating power was also carefully examined, and it was found that whilst with 0.6 per cent. of oxygen the illuminating power is 17 candles, when 2½, 3, and 5 per cent. of air are added instead, the illuminating power falls to 13.45, 13.04, and 10.09 candles respectively.

Comparative experiments with 3 per cent. of air in the one case and 0.6 per cent. of oxygen in the other, proved beyond doubt the value of oxygen over any possible system of air purification, in economy of space, lime, and labour, although an explanation of the cause of this difference cannot yet be given. Purifiers which, when pure oxygen was used, reduced the percentage of sulphur enormously, ceased to so reduce it directly air containing the same percentage of oxygen was used. And after a short time they actually let the gas issue more impregnated with sulphur compounds than it was when it entered the purifiers; and when the air was stopped and pure oxygen again admitted, the bad effect immediately ceased, and after an hour or so, when the oxygen had had time to spread through the material, the purifiers began to do good work again and to reduce the amount of sulphur.

With regard to the commercial and practical advantages accruing from the use of oxygen, the author asserts that, even where there is unlimited space for purification, or where the works are not under the operation of the Sulphur

Clauses Act, the avoidance of the use of cheap oxygen is an unjustifiable extravagance. The cost of providing, erecting, and maintaining the oxygen plant is discussed in some detail, and the saving from the employment of oxygen is estimated as follows:—The average cost of purification where companies are placed under the Sulphur Clauses Act is 3*l.* 15*s.* per 1,000,000 cubic feet (Field's analysis). The same work in conjunction with oxygen, but not including the cost of the oxygen, is 1*l.* 15*s.* The total cost, except the interest on plant, of 0.6 per cent., or 6,000 cubic feet of oxygen at the higher figure of 2*s.* 6*d.*, would be 15*s.* (a high estimate), and the interest on capital in providing oxygen plant at 10*l.* per million at 10 per cent., 1*l.* Balance in favour of oxygen, 5*s.* If to this sum the interest (3*l.* 15*s.*) on capital due to saving one-half the space and plant required under present system (*i.e.*, 50*l.* per million at 7½ per cent.), and the average cost (6*l.* 5*s.*) of 1½ candles in illuminating power at 1*d.* per candle, are added, the saving per million cubic feet is found to be 10*l.* 5*s.* Even if the interest (3*l.* 15*s.*) on capital saved is not taken into consideration, it still leaves 6*l.* 10*s.* per million to the credit of oxygen.

It is stated in conclusion that the economies of oxygen purification have been designedly understated, the saving claimed having been greatly exceeded at Ramsgate. It is sufficient for the author's purpose to make it clear that the system is self-supporting under all conditions where gas must be purified, and leaving such a margin as to recommend its adoption by all gas companies and corporations. (This Journal, 1889, 82.)—F. S. K.

PATENTS.

An Improvement in Fuel Gas Burners. The Gas Patents Syndicate, Limited. From J. B. Archer, Washington, U.S.A. Eng. Pat. 8560, June 11, 1888. 6*d.*

A HORIZONTAL pipe conveying air is terminated by an elbow piece, through the centre of which a smaller pipe is inserted to convey the fuel gas. A movable plug on the gas pipe serves to spread out the issuing gas, and a globular cap on the air pipe provides for a due admixture of gas and air before leaving the burner. A detailed drawing accompanies the specification.—G. H. B.

Improvements in Gas and Coke Producers and in the Apparatus for Collecting the By-Products therefrom. T. Nicholson, Hucknall Torkard. Eng. Pat. 9239, June 25, 1888. 6*d.*

ABOVE a coal or coke furnace is placed a generator or producer having a perforated bottom and sides. A blast of air is admitted to the furnace below, and the gaseous products from thence play through the perforations in the sides and bottom of the producer upon the charge of fuel within. The gaseous products from the furnace do not rise directly to the producer, but are first conducted to the rear of the apparatus, and are then, for their better distribution, led forward into a heat chamber immediately below the producer. The gas extracted from the charge of fuel in the producer is scrubbed with gas liquor in suitable scrubbers, and then led through a serpentine condenser. Lastly, the coke remaining in the producer is treated with wet steam, which is said to harden it and to remove its nitrogen and sulphur.—A. R. D.

The Generation of Gaseous Fuel and the Combustion thereof in Steam Generator Furnaces. The Gaseous and Liquid Fuel Supply Co., Limited, Manchester, and B. H. Thwaite, Liverpool. Eng. Pat. 10,224, July 14, 1888. 8*d.*

FROM an ordinary boiler flue the firebars are removed, and the fuel is allowed to rest upon a floor of refractory hollow bricks or pipes laid on the bottom of the flue. Below the ordinary door is placed another door for clinkering and other purposes. Attached to the back of this, but at an interval of a few inches from it, is a refractory block through which a mixed blast of air and steam is supplied to the fuel lying on the floor, the few inches of space between the door and the block serving as a distributing chamber

for the said blast. The combustible gases produced by the decomposition of the fuel are met as they pass the furnace bridge by an independent air supply previously heated by traversing channels formed in the hollow floor supporting the fuel. For about 5 feet from its front end the flue is provided with a specially shaped refractory lining, which is raised to incandescence and prevents the deposition of carbon. A nest of open brickwork at the rear end of the flue also becomes highly heated and prevents any combustible gases from passing away unconsumed.—A. R. D.

Improvements in or relating to the Obtaining of Inert Gases. L. Mond and G. Eschellman, Northwich. Eng. Pat. 11,294, August 4, 1888. 4d.

GASES containing oxidising or reducing constituents, or both, are deprived of these by being passed through oxide of iron or manganese of a suitable degree of oxidation heated to from 300° C. to 400° C. The oxide of iron or manganese that has been employed for eliminating an oxidising constituent may be revived for further use by being utilised to remove a reducing gas, or *vice versa*; or the process may be made a continuous one by taking care that gases of opposite tendencies are present in such proportions as to neutralise one another.—A. R. D.

Improvements in Gas-producing Apparatus in which the Carbonisation of Coal is continuously Effected. J. Elliott, Ludlow. Eng. Pat. 11,443, August 8, 1888. 11d.

AN open hopper kept filled with coal is connected by a vertical pipe with the horizontal retort which widens out towards the discharging end to allow for expansion of the coal during carbonisation. The gases are prevented from returning through the hopper by a sliding valve, which is only opened when a charge of coal is let down. The coal is moved forward in the retort by means of a scoop which can be moved forwards and backwards on an arm passing through the retort lid. The coke is received from the discharging end of the retort in a cylinder which revolves air-tight against a suitable face-plate on the retort, and is discharged by rotating the cylinder. The exit for the gas rises from the discharge end of the retort. Means for giving automatic motion to the various parts is described, and a number of retorts may be worked simultaneously. The specification is accompanied by drawings, which should be consulted for details.—G. H. B.

Obtaining Useful Products from Refuse or Residues of Gas Purifying, and Apparatus therefor. C. Dubois, Marseilles, France. Eng. Pat. 7653, May 7, 1889. 6d.

THE old or refuse materials which have been employed in the purification of lighting gas are heated in a rotary retort and at the same time treated with steam, superheated or otherwise. The sulphur is volatilised, and any cyanogen compounds present are decomposed with formation of ammonia. These products are separated and recovered by whatever means may be found most suitable.—A. R. D.

An Improved Apparatus for Manufacturing Carburetted Air Gas. G. P. Armstrong, London. From G. Jaumez, Paris, France. Eng. Pat. 7854, May 10, 1889. 6d.

THIS apparatus is to be worked on the same principle as that patented by Moussard in France, December 27, 1869, No. 88,263. It is claimed, however, to be less complicated, as all the parts are reduced to their simplest form. Liquid hydrocarbon is vaporised by heat and caused to pass through a Giffard's injector, where its expansive force is employed to draw in the air to be carburetted. A gas holder forms part of the apparatus, and the lower portion of this is utilised as a supply cistern for the hydrocarbon. It is situated below the injector or carburettor, so that any condensed liquid may flow back to the cistern. From the body of the holder a pipe passes downwards conducting gas to a burner, over which is placed a lenticular vaporiser. The

hydrocarbon flowing by gravity from the supply cistern above, is here brought to a state of vapour, and passes upwards as such to the carburettor. Leaving this latter mingled with air it goes forward to the holder, and thence through a suitable pressure regulator to be distributed as required.—A. R. D.

Process for Generating Gases known as Water-Gas and Producer-Gas. J. von Langer and L. Cooper, Leeds. Eng. Pat. 8192, May 16, 1889. 4d.

IN the usual process of making water-gas a mass of fuel in a generator is alternately submitted to the action of a blast of hot air from the bottom, and of a blast of steam from the top. The present improved process differs from the above by admitting both blasts at the top of the generator. It is claimed that a much higher proportion of water-gas is obtained and cheaper kinds of fuel can be employed. The same principle of downward direction only is also applicable when producer-gas alone is required, but in this case a reduced quantity of steam is admitted at the top, and a supplementary supply of heated air is introduced half-way down the mass of fuel.—A. R. D.

III.—DESTRUCTIVE DISTILLATION, TAR PRODUCTS, Etc.

New Occurrences of Ozokerite. B. Lach. Chem. Zeit. 13, 831.

OZOKERITE for the cerasin industry is still obtained from Galicia, but new deposits are always being sought for, Ozokerite is known to be found in the Caucasus, and there are reports more or less fanciful from North America of deposits of white ozokerite miles in length being found.

The author having had samples submitted to him, has examined them by his process (Chem. Zeit. 9, 905; this Journal, 1885, 488) with the following results:—

Caucasian ozokerite resembles an inferior Galician variety. Melting point of crude substance 68° C. Treated with 20 per cent. of fuming sulphuric acid it yields 58.1 per cent. of second quality. Cerasin: melting point = 68.5° C. Volatile portion, 1.8 per cent.

The crude substance has a repulsive odour, is worked with difficulty in quantity, becomes stiff in the basin, and presses extremely badly. The price is at present too high, and until greater demand arises and the transportation be accomplished, the Galician ozokerite production will make very strong competition.

North American ozokerite, Colorado. Here extensive mines of an ozokerite-like substance are found, the supply of which is hindered only by the transport difficulty. On being tested, however, it proves useless for the production of cerasin. The material is supplied in cakes 36 × 20 × 6 cm. in dimensions. It is a dull black hard substance, smelling of catechu, and proving brittle and pulverisable. Care must be taken in melting it at 95° C., or violent frothing may occur from the presence of traces of water.

The analysis shows—

Melting point of crude substance 70.0° C.
" " treated " 76.5° C.

Treated by the aforementioned process no cerasin is obtained. Even when heated to 300° C. it will not filter in the slightest through filter-paper, although it is quite a thin fluid. Subjected to distillation it gives the undernoted results:—

	Per Cent. (by difference).
Paraffin and oil.....	80.60
Loss in gas	2.13
Loss in water.....	2.60
Residue	5.28

It commences to distil at 360° C., when nearly 3 per cent. of oil setting at 30° C. comes over. At a much higher temperature it runs over and distils steadily, giving a very fair crude product for paraffin extraction.

Consequently this substance is suitable for paraffin making and is absolutely useless for the cerasin industry.

The price is at present disproportionately high.—D. A. S.

Recovery of Tar and Ammonia in the Manufacture of Coke. Stahl. u. Eisen, 1889, 9, 482.

GREAT progress has been made in plant for condensing the tar and ammonia formed in the manufacture of coke, and it has been found that by condensing it the quality of the coke has not deteriorated. The Hoffmann-Otto ovens have proved excellent, and bee-hive ovens have also been used. In the water condensers not only the fluid tar but also much viscid tar is deposited, so it is advisable to make use first of air condensers which consist of large chambers with sheet-metal partitions between which the gases have to travel zig-zag, and thus deposit most of the tar and coal dust. After leaving the water-condensers the gases are passed through washers to remove the remainder of the tarry products. Since only 11 to 14 per cent. of the nitrogen in the coal are converted into ammonia, numerous attempts have been made to increase the yield. It has been proved in practice that instead of being diminished, as hitherto supposed, the yield is increased by employing a higher temperature, and the introduction of hydrogen into the coke ovens still further increases the yield. As hydrogen is too expensive, dissociation of water in the ovens has been tried, but the loss of coke in consequence is too great. The use of lime is also unsatisfactory, as the quality of the coke is deteriorated.

A greater yield of tar can be obtained by keeping the upper part of the ovens cool, but its quality is deteriorated owing to the decrease in the light oils. The tar is sent away for distillation, but the ammonia liquors are almost always worked up at the coke factories, generally into ammonium sulphate.—E. E. B.

Discovery of New Petroleum Wells near Baku.

A REPORT, dated the 22nd August last, has been received at the Foreign Office from P. Stephens, British Vice-Consul at Batoum, stating that news had been received in Batoum on the previous day to the effect that eight new fountains had been discovered in the naphtha-producing territory of that district, the combined output of which, at the present rate of supply, could approximately be estimated at 16,000 tons per diem, in consequence of which the price of crude oil at Baku had decreased from $\frac{1}{2}$ d. to $\frac{3}{4}$ d. per gallon, and a further fall was expected.

"Peat" Candles in South America.

"PEAT" candles are not made of peat, but of the paraffin that is distilled from peat in Brazil, where it is more plentiful even than in Ireland. According to *The Comercio des Amazonas*, John Grant and Co. are now having machinery installed at their works capable of producing 80 tons of peat paraffin a month. They are also extracting from the turf a valuable lubricating oil. They employ 300 workmen, and have 33 boilers with purifiers, stills, refrigerators in their plant.—*Engineering and Mining Journal*.

Petroleum in the Canadian North West.

THE *Canadian Gazette*, in its issue of August 8th, publishes the following:—

Interesting particulars of the rich find of oil in the North-West reached Ottawa on July 23rd in a letter received by Alfred Patrick, late clerk of the Dominion House of Commons, from his son, Major Allan Poyntz Patrick, Dominion Topographical Surveyor, of Calgary. The letter says:—"For 11 weeks we have been prospecting among the mountains for coal-oil deposits. I am happy to say we have been eminently successful. We have located a

considerable quantity of oil land, some on the eastern slope, and some on the British Columbia side. So soon as we felt perfectly certain as to the importance of our find we had several samples assayed. The result gave 91 per cent. lubricating oil, 5 per cent. heavy oil, 1 per cent. water, and 3 per cent. foreign matter. This is the best oil ever discovered, and far ahead of the Pennsylvanian oil-fields. There is plenty of head, the flow being about 4,500 feet above the sea level, and the crude oil can be run off in pipes any distance. It is the greatest find ever known. All the oil lands are within easy distance from the railway." The letter further states that arrangements are being made for developing the find, which, if correctly reported, adds immensely to the treasure to be expected from the North-West.

PATENTS.

Improvements in and Relating to the Manufacture of Acetic Acid and its By-products. R. Weiss, Zossen, Germany. Eng. Pat. 9835, July 6, 1888. 6d.

THESE improvements refer to the distillation of wood, peat, brown coal, or other organic substances, for obtaining carbon and various volatile products, notably acetic acid. It is proposed to introduce into the retorts a current of heated water or generator gas, or in fact any gas free from oxygen and containing carbonic oxide. This materially aids the evolution of the volatile products, and is said to largely increase their yield by removing them quickly from contact with the residual heated carbon, which is apt to promote their decomposition.—A. R. D.

Improvements in Vertical or Inclined Retorts for Distilling Shale, Coal, and other Analogous Substances. J. Jones, Dalmeny. Eng. Pat. 9893, July 7, 1888. 8d.

IN ORDER to facilitate the discharge of the contents of the retort, the lower part is built so as to incline downwards to the discharge orifice, which may be closed by a sliding door as well as by an ordinary retort door. A suitable motion of a shovel applied at the bottom of a retort of this construction will cause the whole contents to descend evenly through the retort. A further claim refers to the employment of fireclay blocks having a feather and groove joint all round the block, of which the retort is to be constructed. The specification is accompanied by drawings.—G. H. B.

Improved Coating of Preservative Material for Iron, Steel, and other Structures, or for Coating the Bottoms of Iron and Steel Ships or other Vessels, and Process of Manufacturing the same and other Products from Gas Tar. J. H. Eastman, Liverpool. Eng. Pat. 10,192, July 13, 1888. 4d.

See under XIII., page 715.

IV.—COLOURING MATTERS AND DYES.

On Mononitroso- and Dinitrosomaphthoresorcinol. S. v. Kostanecki. Ber. 22, 1342—1347.

NEITHER the resorcinol of the naphthalene series nor its derivatives have been hitherto known. The author describes some compounds obtained from hydroxymaphthoquinone, $C_{10}H_5O_2.OH(O : O : OH = 1 : 4 : 2)$, belonging to this class of bodies.

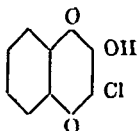
Hydroxynaphthoquinonemonoxime, $C_{10}H_5(O.NOH)OH$, *Mononitrosomaphthoresorcinol* is obtained by treating hydroxynaphthoquinone (1 mol.) dissolved in caustic soda (2 mols.) with a solution of hydroxylamine hydrochloride (1 mol.). The yellowish-red solution turns to an olive-green, and on addition of acid a light yellow precipitate is formed, possessing all the properties of a nitrosophenol. Recrystallised from glacial acetic acid it forms yellowish

needles, decomposing at 180° C. with evolution of gas. To obtain a pure product it is essential to work with reagents free from iron. This body is practically insoluble in cold or hot water and ether, easily soluble in alcohol, caustic soda and carbonate of soda. Ferrous salts produce in its alcoholic solution a green lake, ferric salts a dark brown one. Cobalt salts produce a dark yellow; nickel salts a scarlet red precipitate. The mononitrosomaphthoresorcinol dyes mordanted fibres. Olive-green colours are obtained with iron mordants and scarlets with nickel mordants. The latter are, however, not fast to boiling soap solutions.

Monochloromononitrosomaphthoresorcinol—

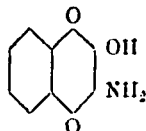


Chloronaphthalenic acid—



is acted upon by hydroxylamine in the same way as hydroxynaphthoquinone, yielding finally a chlorinated product which, after recrystallisation from glacial acetic acid, forms yellow needles, decomposing at 178° C. It dyes mordanted fibre.

Dinitrosomaphthoresorcinol, $C_{10}H_7(O.NOH)_2$.—If one molecule of the mononitroso-compound and one molecule of sodium nitrite be dissolved in dilute caustic alkali and poured into dilute acid, a light yellow crystalline precipitate is formed, which after recrystallisation from alcohol, forms long plates nearly white and decomposing at 165°. It crystallises with one molecule of water and dyes mordanted fibre darker than the mononitroso-compound. On oxidation with nitric acid phthalic acid is formed. On reduction with tin and hydrochloric acid Merz and Weith's naphthalenic acid—

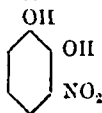


was obtained.—A. L.

On Colouring Matters which Dye Mordanted Fibres.

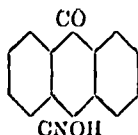
St. v. Kostanecki. Ber. 22, 1347—1353.

COLOURING matters having the character of phenols dye fibres mordanted with oxides, if two hydroxyl-groups are in the ortho-position. As a further instance that this theory is correct, the volatile nitropyrocatechin (nitrocatechol)—



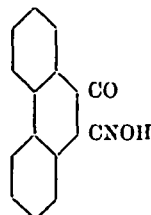
was examined, the result being that one more proof for this theory was obtained. It produces orange shades with aluminium mordants, whilst its non-volatile isomeride yields a light yellow under the same conditions. Nitroso-phenols act in the same way if they are orthoquinone-oximes. Several similar compounds were examined.

Anthraquinone-oxime—



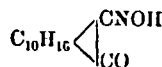
can be considered a para-nitrosophenol, and does not form dyes with metallic oxides.

Phenanthrenequinone-oxime—



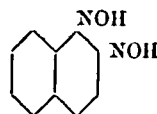
is an ortho-compound, and acts as a dye towards fibres mordanted with oxides.

Retenequinone-oxime—



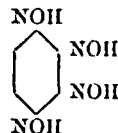
produces green shades with iron mordants.

Orthoquinonedioximes form dyes with oxide mordants.
—*Orthonaphthalenedioxime—*



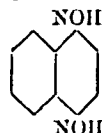
produces brown shades with iron or cobalt mordants.

Orthobenzenetetroxime—



forms dark browns with iron mordants.

The isomeric para-compounds, as *p*-naphthalenedioxime—



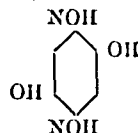
and

p-benzenedioxime—



do not form dyes with oxide mordants.

Orthohydroxyoximes form dyes with oxide mordants.
The dioxime of dihydroxyquinone—

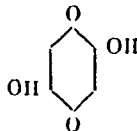


was proved to be a colouring matter in that sense as well as the hydroxynaphthoquinone-oxime, whilst the isomeride of juglonemoxime, which is not an ortho-compound, does not act as a dye.

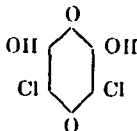
Orthohydroxyquinones are not all dyestuffs. Experiments with hydroxythymoquinone proved a failure, whilst the tests with *o*-hydroxynaphthoquinone were successful,

as well as those made with Mylius' hydroxyjuglone. Far stronger colours are, however, obtained with such bodies, which contain this complex of atoms twice.

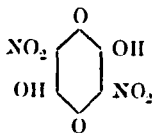
Dihydroxyquinone—



Chloranilic acid—

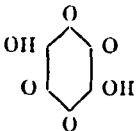


Nitrilanilic acid—



and

Rhodizonic acid



are all capable of dyeing upon oxide mordants.—A. L.

On Para-Diamidodiphenylpiperazine and the Formation of Colouring Matters from it. E. Lellmann and C. Schleich. Ber. 22, 1387—1389.

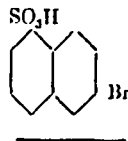
PARAMIDOPHENYLPIPERIDINE forms colouring matters, analogous to those obtained from paramidodimethylaniline. The authors examined the *p*-diamidodiphenylpiperazine in the same direction. It was prepared by heating 100 grms. of aniline with 100 grms. of ethylene bromide on the water-bath; a violent reaction sets in and the whole mass solidifies. Then 110 grms. of ethylenebromide were added and the mass heated to 130°—140° C. in the air-bath, whilst as much caustic potash was gradually added as would saturate the whole of the bromine. After several hours' boiling the organic compounds solidify. After washing with water they are distilled with steam to remove the excess of ethylene bromide and then treated with hot alcohol, by which the impurities are dissolved. The yield from 100 grms. of aniline was 90—100 of pure base. The nitrosation was performed in the same way as Morley describes, and the nitroso product without further purification was reduced with stannous chloride and hydrochloric acid. After precipitating the tin by sulphuretted hydrogen the hydrochloride of the diamido base was obtained in colourless prisms, crystallising with 4 mols. of water, $C_{16}H_{20}N_4(HCl)_4 + 4 H_2O$. On oxidation with 2 mols. of phenol or naphthol by ferric chloride, blue colouring matters of the indophenol group were obtained. If the phenol be replaced by the hydrochloride of an amine and the oxidation be performed by potassium bichromate, indamines are formed, of which the one prepared from aniline can be converted after addition of two more molecules of aniline hydrochloride in acetic acid solution, into a safranine. With metatolylenediamine a difficultly soluble blue colouring matter is formed, which is analogous to Toluylene blue, and is changed into a red colouring matter on boiling its hydrochloric acid solution. Dyestuffs similar to Methylene blue could not be prepared. On oxidation with sulphuretted hydrogen and ferric chloride a red colour was obtained, but no blue.—A. L.

On two β -Bromonaphthalenesulphonic Acids. S. Forsling. Ber. 22, 1400—1403.

THE author obtained these two acids by converting Brönnner's β -naphthylaminesulphonic acid and Dahl's β -naphthylaminesulphonic acid into the corresponding diazo-compounds and treating these with fuming hydrobromic acid and cuprous bromide. The concentrated solutions were neutralised with potassium carbonate, when the potassium salts were precipitated.

$\beta_1 = \beta_3$ -Bromonaphthalenesulphonic acid obtained from Brönnner's naphthylaminesulphonic acid is very soluble in water. Its salts are, however, very difficultly soluble in it. The potassium, ammonium, and barium salts are described. The bromide of this acid was prepared in the usual way with phosphorus pentabromide. It is easily soluble in benzene and chloroform, with difficulty in petroleum spirit and ether. It forms prisms melting at 110° C. By boiling it with alcoholic ammonia the amide is formed, crystallising in fine long needles, melting at 207° C. On distilling the bromide with the calculated amount of phosphorus pentabromide, dibromonaphthalene is obtained, which, after recrystallisation from alcohol and distillation with steam, was obtained from its solution in ether and chloroform, in colourless square plates, melting at 158° C.

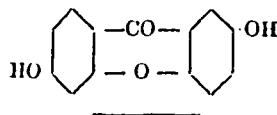
$\beta_1 = \alpha_5$ -bromonaphthalenesulphonic acid was obtained from Dahl's naphthylaminesulphonic acid; its bromide, prepared in the usual way, forms broad needles, melting at 151° C. It was converted into dibromonaphthalene, crystallising in white needles, melting at 75° C. It is identical with the compound obtained by Meldola and Darmstädter, and Wichelhaus, and its constitution is therefore—



—A. L.

Synthesis of Euxanthone. C. Gräbe. Ber. 22, 1405—1406.

ALL attempts at a synthesis of euxanthone have hitherto failed, only isomeric compounds having been obtained. The author has, however, at last succeeded in preparing this body by the action of anhydrous acetic acid on a mixture of β -resorcylic acid and hydroquinone carbonic acid, and by distillation of the product thus obtained. It is in every respect identical with euxanthone; its acetyl compounds have the same properties, and its constitution is therefore—



—A. L.

Studies on Brazilin. C. Schall and C. Dralle. Ber. 22, 1547—1564.

NITRIC acid produces with brazilin tetramethylether first a blood-red, then a brownish-red, and finally an olive-green colouration. An estimation of the molecular weight by Raoult's method proved that the formula $C_{16}H_{16}O(OC_2H_5)_4$ is consistent therewith. By the action of bromine, derivatives are obtained, which do not lose any bromine on treatment with dilute ammonia, and which can be crystallised, whilst others losing bromine under the same treatment, crystallise from acetic acid. The analysis of the latter bodies was carried out in the following manner:—

The material was dried over zinc chloride or calcium hydrate until no further loss of weight was observed. The bromine was estimated by treatment with dilute ammonia. Bromine escaping with acetic acid was absorbed in dilute ammonia.

Method of Decomposition.—The weighed substance was decomposed at 40° to 50° C. by standard ammonia, and the excess titrated back with dilute nitric acid, the body to be

analysed itself serving as indicator. It was dried at 195° C. in a stream of dry hydrogen or carbonic acid, the escaping bromine being again absorbed in ammonia, and after deducting its weight from the total loss of weight, the amount of acetic acid was obtained. Finally the total amount of bromine was estimated and a combustion was made.

A.—Derivatives not yielding up Bromine to dilute Ammonia.

Monobromobrazilin was obtained by Buehka and Erk by the action of bromine on tetracetylbraziln and by removing the acetyl groups afterwards. The authors prepared a monobromo product from the tetramethylether. After converting Buehka and Erk's substance into the tetramethylether, its melting point was observed to be 2° — 3° C. lower than the one prepared by the authors. A further examination will decide whether the compounds are isomerides or not.

Dibromobrazilin was prepared by the action of 3 mols. of bromine on 1 mol. of brazilin in glacial acetic acid for several days at the ordinary temperature, until the cherry-red solution turns light brown and dibromobrazilin crystallises out. From the mother-liquors dibromobrazilin is obtained in light red plates, which for further purification were recrystallised from water with addition of sulphurous acid. White plates, melting between 170° — 180° C. to a ruby-red mass, were obtained. With anhydrous acetic acid a tetracetyl compound is formed, melting after several recrystallisations at 185° C.

Tribromobrazilin.—3 mols. of bromine dissolved in glacial acetic acid are allowed to act for one hour on 1 mol. of brazilin in glacial acetic acid solution. The mixture is poured into cold water and the orange precipitate recrystallised from dilute alcohol with addition of sulphurous acid. It turns brown at 197° — 200° C. without melting. Its tri-acetyl compound melts at 195° C. and its tetracetyl compound at 263° C.

B.—Derivatives losing Bromine on treatment with dilute Ammonia.

Tetrabromobrazilin (tribromobrazilinmonobromide) is obtained by boiling 2 or 3 mols. of bromine with 1 mol. of brazilin in glacial acetic acid solution. The yield is from 14 to 15 per cent. It loses one atom of bromine on treatment with dilute ammonia. The complete analysis leaves it undecided whether $C_{16}H_5Br_4O_5 + 1\frac{1}{2}$ aq. or $C_{16}H_6Br_4O_5 + 2$ aq. represents the correct formula.

Hexabromobrazilin (tribromobrazilintribromide).—5 grms. of brazilin dissolved in 100 grms. of glacial acetic acid are filtered through glass wool, and a solution of 25 grms. of bromine in 25 grms. of glacial acetic acid is quickly added. The mixture is boiled for half a minute, and on cooling an abundant crystallisation sets in—large brownish-red crystals—losing bromine at 170° — 180° C. The substance crystallised from perchloro-ethylene; water or alcohol decomposed it, hydrobromic acid being formed. Its formula is $C_{16}H_6Br_6O_5 + 2$ aq.

Octobromobrazilin (tetrabromobrazilintetrabromide) is obtained like the hexa-compound, only the amount of bromine used is doubled. It loses bromine between 130° — 140° C. Formula $C_{20}H_{12}Br_8O_9 + 2$ aq.

Nonobromobrazilin (tetrabromobrazilinpentabromide) is obtained like the former compound, if the mixture of bromine and brazilin be boiled for 15 minutes instead of one minute. Formula $C_{16}H_5Br_9O_5 + 2$ aq.

It is a remarkable fact that on drying the bodies of this series bromine always escapes as hydrobromic acid and not as free bromine.

Among the oxidation products the authors discovered a resorcinol carbonic acid. The authors further studied the properties of brazilin obtained according to Erdmann and Schulze's method and according to Buehka and Erk's method. They prepared the reduction products, the acetyl derivatives produced by acetyl chloride and anhydrous acetic acid.

The brazilin tetramethyl ether, as also the new trimethyl ether demonstrates, along with the acetyl compounds, the phenolic nature of the four brazilin oxygen atoms. Two monobromobrazilins have been in all probability identified, besides the two dibromo-compounds, and a new tribromobrazilin. All these are colourless substances. But so soon as bromine is introduced in an unstable position, from which it is readily eliminated again, then derivatives possessing tinctorial powers arise.—A. L.

PATENTS.

Manufacture of Sulpho-Acids, and Colouring Matters therefrom. F. Wirth, Frankfort-on-the-Maine, Germany. From "The Farbenfabrik vormals Brömer," Frankfort-on-the-Maine, Germany. Eng. Pat. 3724, August 4, 1882. (Second Edition.) 6d.

WHEN naphthol sulphonic acids or their salts are treated with ammonia at a high temperature they are converted into the corresponding naphthylamine sulphonic acids. If β -naphthol be heated with twice its weight of concentrated sulphuric acid of 66° B. for several hours on a water-bath, it is converted into β -naphthol monosulphonic acid. 60 kilos. of the neutral or acid ammonium salt of this acid are heated for 24 hours to 150° — 180° C., together with 60 kilos. of water and 12 kilos. of calcium hydrate under pressure. The melt is dissolved in hot water, filtered, diluted with about 500 litres of water and made acid. The β -naphthylamine sulphonic acid at once separates, and can be purified by redissolving in an alkali and precipitating with an acid. The acid is slightly soluble in hot water, and almost insoluble in cold, and separates in glistening flakey crystals. Its barium salt crystallises in needles containing 5 molecules of water. The diazo-compound forms colouring matters with the phenols and their derivatives, giving yellows to brownish reds. The combination with resorcinol yields a golden yellow dyestuff, and α -naphthol or its sulphonic acid yields a blood-red dye. The mono- and disulphonic acids of both naphthols can be converted in a similar manner. According to the patent, another β -naphthylamine sulphonic acid is obtained by heating under pressure, at about 180° , 50 kilos. of ammonium β -naphthol sulphonate, 25 kilos. of calcined sodium carbonate of 98°, together with water. The acid formed is almost insoluble in alcohol, but easily soluble in water, whereas the sodium salt is readily soluble in alcohol. The diazo-compound combined with α -naphthol produces a bluish-red dyestuff.—T. A. L.

The Production of New Azo-Colours. M. Hoffmann and A. Weinberg, Mainkur, Germany. Eng. Pat. 9214, July 31, 1885. (Amended, June 15, 1889.) 4d.

THE diazo-compounds of the sulphonic acids of α - and β -naphthylamine combine with α -naphthylamine to form azo-compounds, and these bodies may be again diazotised and recombined with the naphthols or naphthol sulphonic acids in an alkaline solution. The following is the method employed for working the invention:—35 kilos. of sodium α -naphthylamine disulphonate are dissolved in 300 litres of water and 30 kilos. of hydrochloric acid of 21° B., and diazotised by means of 7 kilos. of sodium nitrite dissolved in water. 18 kilos. of α -naphthylamine hydrochloride dissolved in 500 litres of water are then added under constant stirring. A dark violet precipitate separates, and the whole, after standing 12 hours, is treated with 12 kilos. of hydrochloric acid and 7 kilos. of sodium nitrite. The diazo-azo-compound thus formed is added to a solution of 36 kilos. of sodium β -naphthol- α -disulphonate, which is kept alkaline by the addition of 20 kilos. of ammonia of 25 per cent. The colouring matter which is immediately formed is salted out, filter-pressed and dried. Other α - or β -naphthylamine sulphonic acids may be used in place of the α -naphthylamine disulphonic acid mentioned above, but no claim is made to the use of Piria's naphthionic acid or to other amido-mono-sulphonic acids (i.e., those which are not naphthalene derivatives). Other mono- and disulphonic acids of the naphthols, or the naphthols themselves, may be employed in place of

the β -naphthol- α -disulphonic acid mentioned in the second stage of the process described above. The shades given by the various combinations vary from reddish-violet to dark blue.—T. A. L.

Process for the Production of Mixed Azo-Colours.
P. Jensen, London. From the "Actiengesellschaft für Anilinfabrikation," Berlin, Germany. Eng. Pat. 15,296, December 12, 1885. (Second Edition.) 6d.

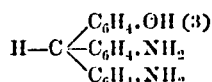
ALREADY described in this Journal, 1886, 595.—T. A. L.

Improvements in the Production of Mixed Azo-Colours.
C. A. Martius, Berlin, Germany. Eng. Pat. 2213, February 15, 1886. (Second Edition.) 6d.

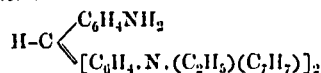
This patent is an extension of the preceding one (this Journal 1887, 138), and enumerates a number of amines and phenols together with their sulphonic and carboxylic acids with which tetrazo-diphenyl and tetrazo-ditolyl may be combined, producing mixed azo-colours. Examples are also given relating to the employment of benzidine carboxylic acid and benzidine disulphonic acid, together with their homologues, in the formation of colouring matters.—T. A. L.

Improvements in the Production of Green and Bluish-Green Colouring Matters. O. Imray, London. From the "Farbwerke vormals Meister, Lucius and Brüning," Höchst-on-the-Maine, Germany. Eng. Pat. 12,796, September 4, 1888. 8d.

This invention describes the production of green and bluish-green colouring matters which are to be considered as derivatives of *m*-hydroxydiamidodiphenylmethane—



The leuco-bases of these colouring matters are produced either by condensing *m*-nitrobenzaldehyde with tertiary aromatic amines (Ber. 12, 802), reducing and converting the amines into the corresponding hydroxy-compounds by the action of nitrous acid, or by condensing directly *m*-hydroxybenzaldehyde with tertiary aromatic amines. Colouring matters are produced from these leuco-bases by suitable oxidising agents, and are then converted into sulphonic acids, or the leuco-bases are sulphonated and the leucosulphonic acids subsequently oxidised. The patent also describes the compounds obtained from the methoxy- and ethoxy-derivatives. It is said that the sulphonic acids of the colouring matters when dyed are specially fast to light and washing. The formation of the oxy-compounds from the amido-compounds is illustrated by the following example: 50 parts by weight of *m*-amidotetraethylamidodiphenylmethane are dissolved in 35 parts by weight of hydrochloric acid of 33 per cent., and 5,000 parts by weight of water, the whole cooled to 0° and a solution of 8.9 parts of sodium nitrite of 96.5 per cent. slowly added. The whole is then warmed to 60° C. for some time, and finally boiled until nitrogen is no longer evolved. Soda lye is then added till alkaline, when the *m*-hydroxy-leuco-base is precipitated as a crystalline powder which is filtered off, boiled, and dried. In the case of bodies having less basic properties the method is slightly modified. Thirty-five parts by weight of *m*-amidodibenzyl-diethylamidodiphenylmethane—



are dissolved in 30 parts by weight of concentrated sulphuric acid and 4,000 parts of water, cooled to 0° and diazotised with 4.7 parts of sodium nitrite of 96.6 per cent. dissolved in water, the decomposition of the diazo-compound being carried out as described above. These hydroxy-compounds may also be obtained by direct condensation. Twenty

parts of diethylaniline, eight parts of concentrated sulphuric acid, and nine parts of *m*-hydroxybenzaldehyde are heated in a lead-lined boiler fitted with an agitator to 130° C. for 72 hours, the mass made alkaline with soda lye, the unaltered diethylaniline distilled off with steam, and the hydroxy-leuco-base remaining repeatedly boiled with water and dried. The *m*-hydroxy-leuco-bases prepared according to either of the preceding processes may be oxidised as follows into the corresponding colouring matters. Ten parts of *m*-hydroxy-tetra-ethylamidodiphenylmethane are dissolved in 8.8 parts of hydrochloric acid of 30.8 per cent. and the calculated quantity of manganese or lead peroxide suspended in water is added. The colouring matter is precipitated from the filtered solution by the addition of salt and purified by recrystallisation. The hydrochloride of the base thus obtained forms brilliant needles of the colour of cantharides and dyes wool, silk, and tanned cotton a yellowish-green shade. These bodies may be converted into "acid colouring matters" by heating them with six times their weight of fuming sulphuric acid to 50°–60° C. until a test dissolves in dilute ammonia with a blue colour. The products are identical with those obtained by sulphonating the hydroxy-leuco-bases and subsequent oxidation as follows: 30 parts of *m*-hydroxytetraethylamidodiphenylmethane are dissolved in 150 parts of fuming sulphuric acid containing 10 per cent. SO_3 , and the solution allowed to stand until a sample gives a clear solution with dilute ammonia. In the case of some of the compounds it may be necessary to use a stronger sulphuric acid, and to complete the reaction by heating to 70° C. In either case the product of the reaction is poured into water and converted into the calcium salt. To prepare colouring matters from these; 10 parts of the calcium sulphonate from *m*-hydroxytetra-ethylamidodiphenylmethane are dissolved in 300 parts of water and oxidised by adding the calculated amounts of dilute sulphuric acid and lead peroxide filtered, and the blue solution of the colouring matter evaporated to dryness. These colouring matters form copper-red powders of a metallic lustre, dissolving easily in water with a blue colour. Excess of a mineral acid turns the aqueous solution green, but the solution is unaffected even when boiled with ammonia or soda, and turns violet only when boiled with soda lye. They dye wool and silk very even bluish-green tints. Colouring matters belonging to the same series are obtained in a manner similar to the processes described above, either by condensing *m*-methoxy- or ethoxybenzaldehyde with tertiary amines or by methylating or ethylating the hydroxy-leuco-bases or the calcium salts of their sulphonic acids mentioned above, and sulphonating and oxidising the compounds so formed in the manner already described. These ethers are said to give better and bluer tints, and are quite as fast as the now substituted hydroxy-derivatives.—T. A. L.

Production of New Colouring Matters. T. R. Shillito, London. From J. R. Geigy, Basle, Switzerland. Eng. Pat. 13,767, September 24, 1888. 6d.

Azoxy and azo-compounds are formed by heating aromatic nitro-compounds with aromatic amines in presence of caustic alkalis. 10 kilos. of *o*-toluidine are heated with 10 kilos. of caustic alkali, and 10 kilos. of nitrobenzene are gradually added. A reddish-brown paste results and the reaction is complete so soon as the mass no longer smells of nitrobenzene. It is then diluted with water, boiled with zinc dust until decolorised, filtered and poured into concentrated hydrochloric acid, boiled and the diamidophenyltolyl formed is precipitated by adding sodium sulphate. The new *p*-diamine formed closely resembles benzidine and toluidine, and is the next higher homologue of benzidine and the next lower one to toluidine. Its sulphate is sparingly soluble. When diazotised it can be combined with amines and phenols similar to benzidine and the colouring matters thus formed when rendered soluble by sulphonation dye unmoderated cotton yellow and red to violet shades.

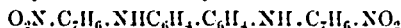
—T. A. L.

Production of Yellow, Orange, and Brown Colours of Alizarin-like Properties. T. R. Shillito, London. From J. R. Geigy, Basle, Switzerland. Eng. Pat. 13,920, September 27, 1888. 4d.

THESE colouring matters are produced by the action of nitric acid on the bodies obtained by combining diazo-compounds with aromatic hydroxy-acids. 12 kilos. of aniline or a corresponding quantity of toluidine or naphthylamine are dissolved in 36 kilos. of hydrochloric acid and 300 litres of water and diazotised with 9 kilos. of sodium nitrite at 5° C. The solution of the diazo-chloride thus obtained is poured into an alkaline solution of 17 kilos. of salicylic acid or the corresponding quantity of any other oxy-carboxylic acid, and the dyestuff precipitated with salt or an acid, filter-pressed and dried. 45 kilos. of the dry compound are dissolved in 135 kilos. of sulphuric acid and a mixture of 20 kilos. of nitric acid and 15 kilos. of sulphuric acid is slowly added. After some time the whole is poured into water, filtered, washed, and brought to the form of a paste. —T. A. L.

The Production of Dinitrodibenzylbenzidine and Dinitrodibenzyltolidine and Transformation of the same into Diamidodibenzylbenzidine and Diamidodibenzyltolidine respectively. A. Bang, Leeds. From Dahl and Co., Barmen, Germany. Eng. Pat. 3628, March 1, 1889. 6d.

By heating 1 molecule of benzidine with 2 molecules of *p*-nitrobenzylchloride the compound—



is obtained, which on reduction yields the corresponding amido-compound. This by the action of nitrous acid gives tetrazo-compounds, which when combined with amines and phenols or their sulphonic acids yield new colouring matters dyeing cotton yellow shades than those derived from benzidine or toluidine. 18.4 kilos. of benzidine are heated for 3—4 days to 100° C. with 34 kilos. of *p*-nitrobenzylchloride (according to the claim "in water suspension") in an enamelled boiler provided with an agitator and condenser. The dinitrodibenzylbenzidine separates as a fine powder, and after filtering off is boiled twice with dilute hydrochloric acid in order to remove unaltered benzidine, after which it is reduced with 35 kilos. of tin and 15 kilos. of hydrochloric acid. The whole is then diluted with water, filtered, and the tin precipitated by means of zinc. The solution containing diamidodibenzylbenzidine and zinc chloride may then be directly diazotised and used for the production of dyestuffs. Diamidodibenzylbenzidine is an amorphous powder scarcely soluble in water but tolerably soluble in alcohol. Its chloride is very easily soluble in water, and the solution is not precipitated by sulphuric acid. Sulphuric acid, however, gives a precipitate when added to an alcoholic solution of the base. The tolidine derivative has similar properties.—T. A. L.

Improvements in the Production of Colouring Matter from Coal-Tar Products. H. D. Kendall, Lowell, Massachusetts, U.S.A. Eng. Pat. 9384, June 6, 1889. 4d.

THIS invention describes the production of a new fast brown colouring matter called "Essacine," by the action of sodium hydrosulphite on dinitrosoresorcinol or its homologues. 22 lb. of zinc are digested for some time with 17 gallons of sodium bisulphite solution of 35° B., and 1 part by weight of this solution is added to 3 parts by weight of commercial dinitrosoresorcinol containing about 25 per cent. of water. The mixture is constantly agitated when the temperature rises to about 57° C. and the reaction is completed in about one hour. The resulting liquid contains the essacine and may be diluted as desired. Cotton requires to be mordanted, preferably with chromium, the shades produced being, according to the patent, absolutely fast to soap, light, and acids. It can also be dyed together with alizarin and when mixed with Coeruleine, Viridine, &c. to produce different shades, gives colours which are said to be faster than when the Essacine is omitted.—T. A. L.

V.—TEXTILES: COTTON, WOOL, SILK, Etc.

Black Spots on Cellulose. W. Herzberg. Mitth. Königl. techn. Versuchs. 1889, 62—64.

A SAMPLE of cellulose prepared by the Ritter-Kellner process was disfigured by numerous black spots varying in size from that of a pin head to that of a medium-sized pea.

The appearance was quite different from that occurring in straw cellulose which has been stored in damp places. Microscopical examination demonstrated the existence of a fungoid growth, twining round the cellulose fibres as ivy does round a tree. The brown colour of its mycelium caused the patches of it to be visible to the naked eye as dark specks. It was thought that the germs had been derived from the river water used in the manufacture, spring water not being available, but it is more likely that they came from the air, finding a good soil on the moist cellulose.

Calcium sulphite was recognised on the spots by Frank's method with iodine solution (*Papier-Zeitung*, 1888, 10), and if this be viewed as the cause of the growth the obvious remedy is to avoid its presence in the finished product; on the other hand the acid juices of the growth itself will tend to liberate sulphurous acid from the calcium sulphite and arrest its development. Thorough drying is an effective preventative, and where this is impracticable zinc chloride in the proportion of 1 litre at 40° B. to 100 litres of water proves a reliable antiseptic, 40 kilos. of such a solution sufficing for 100,000 kilos. of cellulose. So small a quantity interferes with neither the sizing nor the colouring processes.

The author recapitulates a few facts bearing on this subject that have appeared from time to time in the "*Papier-Zeitung*."

It was observed that a paper made from pure rags and highly sized with rosin developed a fungoid growth when kept in a warm damp place; there is no direct evidence to show whether the germs are derived from the water or air. Adequate nutriment for the mould is supplied by size of animal origin, and even when rosin is used the accompanying starch may prove sufficient.—B. B.

PATENTS.

Improvements in the Treatment of Flax and other Fibrous Plants for obtaining the Fibre therefrom for Spinning and other Manufacturing Purposes. L. Dekien, Comtrai, Belgium. Eng. Pat. 11,397, August 7, 1888. 6d.

By this invention the costly and troublesome process of retting is entirely done away with and the flax as cut is taken direct from the field to the scutching machine.

—E. J. B.

An Improved Method or Process of Freeing Rhea and other Vegetable Fibres from Cementitious Matter, and Apparatus therefor. G. F. Redfern, London. From E. E. Spence, Rotterdam, Holland. Eng. Pat. 11,973, August 18, 1888. 8d.

THE fibre to be treated is made into a coarse tissue with strong yarn, the latter forming the warp and the former the woof. Two boilers are then filled with this tissue. The boilers are connected together with suitable pipes, so that the liquid used for treating the fibre may be transferred from one to the other at will. In its passage from one boiler to another the liquid passes through a filter-press. A great saving in chemicals is claimed.—E. J. B.

VI.—DYEING, CALICO PRINTING, PAPER STAINING, AND BLEACHING.

The Substantive Cotton-Dyes. E. Noelting. Chem. Zeit. 13, 777—778.

ROSENSTIEHL and Noelting have recently discovered that dimeta-amido-derivatives of azoxy-compounds, such as azoxyaniline (dimeta-amidoazoxybenzene), the two azoxytoluidines, and az-oxymetaxylinine, are bases from which a new class of substantive cotton-dyes may be obtained. The most important of these new dyes is one known as St. Denis red, which is produced by acting with α -naphthol- α -sulphonic acid on diazotised *o*-toluidine. It is distinguished by its fastness and perfect resistance to acids. It is not fixed on the fibre from an alkaline bath so easily as benzopurpurin and Congo-red, but full shades can be produced by modifying the ordinary conditions of dyeing. Rosenstiehl gives the following method of dyeing 10 kilos. of cotton, either yarn or cloth: 200 grms. of St. Denis red are dissolved, by the aid of heat, in 6 litres of caustic soda of 7° B, this solution being poured into a warm bath containing 200 litres of water, 48 kilos. of common salt, and 5 litres of caustic soda of 40° B. The bath is heated to 85°, the cotton entered, the heat raised to 90°—95° and kept at this temperature for half-an-hour. At the end of this time the goods are taken out, squeezed or wrung, and passed for 5—10 minutes into a 1 per cent. solution of sulphuric or hydrochloric acid, then well rinsed and dried. The dye-bath is not exhausted and should be preserved, being made up for use again by the addition of 165 grms. of dyestuff, dissolved in 5.7 litres of caustic soda of 7° B, and 0.5 litre of caustic soda of 40° B. for each 10 kilos. of cotton. A very deep red is obtained by taking, for the same weight of cotton, 300 grms. of dyestuff and 9 litres of caustic soda of 7° B. for the first bath, and 250 grms. of dyestuff with 8½ litres of caustic soda for subsequent baths. The dye-bath should be heated with closed steam and the volume of water evaporated must be replaced. Excellent results are obtained by strictly following the directions.

H. Koechlin recommends, more particularly for piece dyeing, a method based on the fact that the alumina-zinc-magnesia lake of St. Denis red is exceedingly bright and fast to soap. The cloth is mordanted by a passage through a solution containing, per litre of water, either 0.5 litre of acetate of magnesia of 30° B. and 0.5 litre of acetate of alumina, or 150 grms. of sulphate of magnesia and 50 grms. of alum. It is then dried and passed for two minutes into a bath, consisting of 50 grms. of sulphate of zinc and 100 cc. of caustic soda of 38° B. per litre of water, after which the pieces are rinsed and dyed per metre of cloth with 2—3 grms. of dyestuff dissolved in 2½ litres of water along with 400—500 grms. of common salt and 5 grms. of lime, the temperature of the bath being raised to 80° in the course of an hour, maintained at this temperature half an hour, and the cloth then rinsed and dried.

C. Schoen communicates a simpler and quicker method: 35 grms. of dyestuff are dissolved in each litre of water, with the addition of 20 grms. of aluminate of soda of 15° B, and 5 grms. of caustic soda of 38° B. To each litre of this solution there may further be added 100 cc. of a solution prepared by dissolving 400 grms. of sulphate of magnesia per litre of water and adding 200 grms. of caustic soda of 38° B. The pieces are puddled with this solution, left rolled up for an hour, then passed through a bath of 50 grms. of alum per litre of water, rolled up again for two hours, and finally washed and dried. Ammonium chloride or sodium bisulphite may be used instead of alum in the fixing-bath.

Discharge effects may be obtained on the new red by means of stannous salts as with benzopurpurin, &c.—E. B.

Dyeing with Resorcinol Green and Cobalt Nitrate.

E. Kopp. Bull. Soc. Ind. de Rouen, 1889, 17, 139.

THE material is printed with a mixture of cobalt nitrate and sodium acetate in the usual manner, passed through a bath of sodium aluminate or sodium silicate, and then dyed with

resorcinol green (dinitrosoresorcinol), calcium chloride being subsequently added to the bath. Sodium aluminate gives better results than silicate. Dinitrosoresorcinol combines so readily with cobalt oxide that the brown colour, due to the union of these two substances, is produced almost instantaneously.—E. B.

PATENTS.

An Improved Process or Method of Dyeing or Painting on Fabrics and other Materials, and Apparatus employed therefor. C. and H. Dratz, Brussels, Belgium. Eng. Pat. 9044, June 20, 1888. 11d.

THE materials, consisting of textile materials spun, woven, or in fibre, wood, leather, paper, &c., are impregnated with mordants or colours in the form of a spray. Patterns in several colours may be obtained by protecting portions of the material by means of stencil plates or open-work patterns. The mechanical arrangements are described with the aid of drawings.—E. B.

Improvements in Bleaching, Dyeing, &c. J. C. Mewburn. From La Société Leblois, Piceni, et Cie., Paris, France. Eng. Pat. 11,452, August 8, 1888. 8d.

THIS invention has reference to Eng. Pat. 14,637 of 1885 (this Journal, 1886, 533). For bleaching or dyeing without deteriorating or felting the materials, two vessels are employed, one being a vessel which can be hermetically sealed, and in which a vacuum can be produced, the other a vessel in which a wheel revolves. In the vacuum chamber, which is preferably employed for steeping operations, there are placed at the top and bottom perforated pipes for the inlet and discharge of air and liquids, also a rack mounted on wheels, and divided into compartments for the reception of small portable boxes made of perforated sheet metal or formed of hollow rods with jointed sides, which serve to hold the materials to be operated upon. When working, a vacuum is created in the vessel by the upper pipes, and the steeping liquor, which has been prepared in an adjoining vessel, is drawn into the vessel by the pipes at the bottom until the boxes are entirely submerged. The formation of a vacuum is repeated several times, alternating with admissions of air through the lower pipes, which produce an agitation of the bath. The steeping-bath is then emptied, the vessel opened, and the boxes transferred to the compartments of the revolving wheel for the final operations of bleaching or dyeing. The wheel having been charged, the bleaching or dyeing is effected by turning the wheel alternately to the right and left whilst submerged in the bath. The steeping is best done at a temperature of 50° to 30° C. for materials which have to be bleached, and 10° to 80° C. for materials which have to be dyed. In steeping for bleaching, either water alone is used or an alkaline solution, or preferably a solution containing three-tenths per cent. of neutral hypochlorite of soda, made by double decomposition between caustic soda and bleaching powder solution, and known as "chlorogene." The steeping-bath preceding the dyeing may be formed of water or alkaline solution, but preferably contains a little dye, which can serve as a ground for the shade to be afterwards obtained. For certain dyes the preparatory steeping of vegetable fibres may be omitted.

Materials composed of animal fibres, which have already been scoured, are placed in the wheel and bleached or dyed at once, instead of undergoing a preliminary steeping.

—E. B.

Improvements in Dyeing Aniline Black. E. and G. E. Sutcliffe, Halifax. Eng. Pat. 11,806, August 16, 1888. 6d.

IN the dyeing of aniline black there is a formation of precipitate in the bath, not only representing a loss of material, but in the process of dyeing wherein the liquors are caused to circulate, interfering with the dyeing by preventing the flow of liquor. In the method given below, the whole of the dye-liquor employed is absorbed, and relatively less acid and oxidising agent require to be used.

The goods are saturated with a liquor composed of materials in the following proportion:—

120 gallons of a 5 per cent. solution of aniline hydrochloride,
20 gallons of a 5 per cent. solution of potassium chlorate,
15 gallons of a 6 per cent. solution of ferrous sulphate,
4 gallons of commercial hydrochloric acid.

The saturated goods are allowed to rest in a warm room or suitable ageing place for about 24 hours, and then treated according to the stage of development of the black. If the goods at the end of this time are of a green colour they are treated with an oxidising agent, such as bichromate of potassium; if, however, the black has completely developed, they are simply rinsed. Other oxidising agents may be used, such as potassium or sodium bichromate, alkaline chlorates or hypochlorites, bleaching liquor, or any agent ordinarily used for, or which will produce, the desired effect.—E. B.

Improvements in Apparatus for Dyeing, Scouring, &c. W. Brown, Selkirk. Eng. Pat. 12,485, August 30, 1888. 6d.

A VESSEL, serving for the reception of the material to be operated upon, is fitted inside a double-jacketed vessel of similar shape. The inner shell is perforated at the bottom and round the sides near the top. When the apparatus is filled with water, &c. the application of heat causes the liquor to flow through the upper perforations and out at the bottom of the inner shell. In this way the felting of the fibres is avoided. Dyewoods, from which the colouring matter has to be extracted, are, by preference, placed below the inner shell. A drawing is given.—E. B.

Improvement in Blueing Preparations for Laundry and Similar Purposes. H. E. Hounsell, London. From G. A. Conant, Acton, Mass., U.S.A. Eng. Pat. 12,670, September 3, 1888. 6d.

SHEETS of Manilla paper are dipped into a filtered solution of Prussian blue in oxalic acid and dried. Sugar is added to the solution to cause the blue to adhere to the dried paper.—E. B.

An Improved Blue for Laundry Purposes in the Form of Crystals. G. R. B. Kempton, West Ham. Eng. Pat. 5380, March 29, 1889. 4d.

A BLUE aniline or other dye is dissolved in water, and potash alum and common salt mixed with the solution, which is then concentrated and set aside for crystallisation. Blue crystals are thus obtained. It is claimed, as an improvement, for this preparation, that it has an attraction for all fibres, inasmuch as the potash alum acts as a mordant for the same; goods are more readily blueed, and an even colour is produced, as the crystals are perfectly soluble.—E. B.

VII.—ACIDS, ALKALIS, AND SALTS.

Boracite from Douglasshall. C. Oehsenius. Neu. Jahrb. f. Mineralogie, 1889, 1, 271.

CRYSTALS of boracite and iron boracite have been obtained from carnallite at Douglasshall. The more recent layers of carnallite are richer than the older, but the yield is very variable. Pseudomorphs of boracite have been found there, and also in blondite, a mineral similar to glaserite in composition.—E. B. B.

Discoveries at the Saline Deposits at Douglasshall. C. Oehsenius. Neu. Jahrb. f. Mineralogie, 1889, 1, 272.

A. NAUPEIT has found the following minerals in carnallite at Douglasshall:—Crystallised iron-boracite, pyrites, red rock crystal, dark specular iron ore, Löwigite (?) sulphur, Bischofite, and tuchyhydrite.

The so-called "Hartsulze" have also been found associated both with carnallite and kieserite, containing 46 to 58 per cent. magnesium sulphate and about 23 per cent. potassium chloride.

In the kainite is found blædite, krugite (associated with polyhalite), Reichardtite, and blue rock salt.

Another interesting find is a crystallised sulphate of sodium and potassium (glaserite?), which occurs with astrakanite, and contains 59 to 66 per cent. potassium sulphate and 18 to 22 per cent. sodium sulphate.—E. B. B.

Action of Magnesium Chloride on Lead Oxide. A. Voigt. Chem. Zeit. 13, 695.

ANDRÉ has previously described the formation of oxychloride of lead by the action of chlorides of calcium, strontium, and barium on lead plaster, but considered that magnesium chloride had no action. The author, however, finds that upon boiling lead plaster with a concentrated solution of magnesium chloride, filtering, and adding cold water to the filtrate, a precipitate is obtained, which when dried at 100° C. has the formula $3 \text{PbO} \cdot \text{PbCl}_2 \cdot \text{H}_2\text{O}$. Lead carbonate is also soluble in magnesium sodium, though not to the same extent as lead oxide.—E. B. B.

The Removal of Arsenic from Chamber-Acid. Le Roy W. McCay. Chem. Zeit. 13, 725—726.

THE author proposes to remove arsenic from chamber-acid by treating the acid with hydrogen sulphide, whilst it is contained in air-tight leaden cylinders, which are heated by a steam-coil or jacket. The cylinders should also be provided with stirring-gear, to cause the precipitated arsenic pentasulphide to collect together and settle.—E. B.

The Action of Acids, especially of Sulphuric and Hydrochloric Acids, upon Sodium Thiosulphate. W. Vaubel. Ber. 22, 1686—1694.

It is generally assumed that sodium thiosulphate is decomposed by acids with the liberation of sulphur dioxide, the separation of sulphur and the formation of water and of the sodium salt of the acid employed. This is the case with the following acids: formic, acetic, succinic, citric, hydrochloric, hydrobromic, hydriodic, hydrofluoric, nitric, sulphurous, dithionic, dilute sulphuric, and dilute phosphoric. Other acids, however, give rise to the formation of sulphuretted hydrogen. Of these, concentrated sulphuric acid produces the largest proportion of sulphuretted hydrogen, and the quantity increases with the strength of the acid. Aqueous hydrocyanic acid gives, when heated with the solid thiosulphate, a considerable evolution of sulphuretted hydrogen; butyric, oxalic, and tartaric acids cause a slight production of sulphuretted hydrogen, as do hydrofluosilic acid and carbon dioxide. In most of these cases the application of heat favours the formation of the hydrogen sulphide. The author has specially examined the action of sulphuric and of hydrochloric acids.

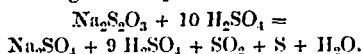
1.—*Decomposition of Sodium Thiosulphate by Sulphuric Acid.*

The decomposition as represented by the following equation is not complete:—



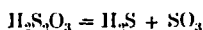
The degree of decomposition varies with the strength of the acid, and in order to obtain concordant results when employing acids of the same strength it is necessary to use 8—10 times as much acid as is required according to the equation. In carrying out the experiments, 10 grms. of the solid thiosulphate were decomposed by the acid in a suitable flask. The decomposition, which was conducted in the cold, was allowed to extend over two hours, when the sulphur dioxide (together with the sulphuretted hydrogen) was determined by absorption with water and titration with iodine. The separated sulphur was collected and weighed, and the total amount of sulphuric acid remaining at the end of the experiment determined volumetrically. The

following table contains the results of the experiments, the percentages being taken on the quantities which should have resulted according to the equation—

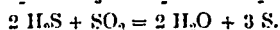
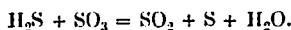


Per Cent. of H_2SO_4 in Acid employed.	SO_2 obtained.	S obtained.	Free H_2SO_4 .
	Per Cent.	Per Cent.	Per Cent.
20	73.9	100.11	98.17
30	82.6	92.21	93.67
40	..	96.71	105.97
50	85.7	104.34	106.01
60	85.3	102.90	107.15
70	84.8	100.60	110.50
80	85.2	95.86	109.60
90	92.0	92.40	109.20
100	45.4	9.15	110.60

In the two last cases sulphuretted hydrogen was formed, which was determined with the sulphur dioxide. In no case does the quantity of sulphur dioxide reach the required amount, whilst that of sulphur exceeds the 100 in four cases, and a steady rise in the percentage of sulphuric acid occurs as the strength of the acid employed in the decomposition increases. It is concluded from this that the thiosulphuric acid at the moment of its liberation decomposes thus :—



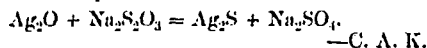
and then that these products mutually react as follows :—



II.—Decomposition of Sodium Thiosulphate by Hydrochloric Acid.

The decomposition in this case supports the above idea as to the decomposition products of thiosulphuric acid. Using 10 times the theoretical amount of 20 per cent. hydrochloric acid under similar conditions to those described above, the quantity of sulphur dioxide was always found to be less, that of sulphur more than should be formed according to theory, and further the production of sulphuric acid was observed in every case.

The action of silver oxide on sodium thiosulphate also confirms the author's view as to the decomposition of thiosulphuric acid. Genthner (Ann. Chem. Pharm. 1884, 226, 232) has shown that silver sulphide and sodium sulphate result, and this decomposition is shown to be a complete one, the total sulphur being in the form of sulphide and sulphate, whilst no sulphurous acid is formed. The following equation represents this decomposition :—



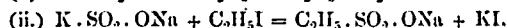
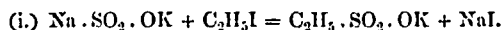
Sulphites and Thiosulphates. A. Schwicker. Ber. 22, 1728—1737.

A SERIES of experiments are described with the view of proving the asymmetric nature of the sulphites and thiosulphates of the alkali metals.

Regarding sulphurous acid as $\text{H}_2\text{SO}_2 \cdot \text{OH}$ two isomeric sodium potassium sulphites $\text{K} \cdot \text{SO}_2 \cdot \text{ONa}$, and $\text{Na} \cdot \text{SO}_2 \cdot \text{OK}$ are possible. By the addition of the requisite quantity of sodium carbonate to a concentrated solution of acid potassium sulphite hard yellowish crystals of the double salt containing two molecules of water of crystallisation result, No. I. Potassium carbonate and acid sodium sulphite give indistinct crystals also of a yellowish colour which contain one molecule of water of crystallisation, No. II.

Heated with ethyl iodide in a sealed tube to 140° , the former gives the double salt $4 \text{C}_2\text{H}_5 \cdot \text{SO}_2 \cdot \text{OK} + \text{NaI}$, the latter $4 \text{C}_2\text{H}_5 \cdot \text{SO}_2 \cdot \text{ONa} + \text{KI}$.

The analytical data agree fairly well with the above composition of these double salts, and it is suggested by the author that in both cases the metal attached to the sulphur has been replaced by the alkyl group according to the equations—



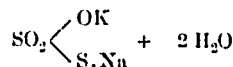
Sodium potassium sulphites corresponding to the above have also been prepared by Röhrig (J. Prakt. Chem. 37, 250; this Journal, 1888, 322).

Neither acid sodium nor potassium sulphite yield double salts with the corresponding normal sulphites. Either of the above sodium potassium sulphites give a double salt with acid sodium sulphite having the composition $\text{HKNa}_2(\text{SO}_3)_2 + 4 \text{H}_2\text{O}$. This salt is also obtained by treating acid sodium sulphite with potassium carbonate, or by evaporating a mixture of the acid sodium and potassium sulphites, or finally by passing sulphur dioxide into a solution of either of the sodium potassium sulphites. In all these cases the products appear to be identical.

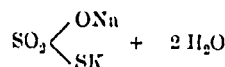
The double salt is stable, and only evolves sulphur dioxide on long standing; it is readily soluble in water, and the concentrated solution combines with acetone, pointing to the presence of an acid sulphite. The double salts $\text{HNaK}_2(\text{SO}_3)_2 + 3 \text{H}_2\text{O}$, and $\text{H(NH}_4\text{)Na}_2(\text{SO}_3)_2 + 4 \text{H}_2\text{O}$ are also described; they resemble the above in properties, and the latter is obtained by passing ammonia gas into a concentrated solution of acid sodium sulphite.

The sodium potassium sulphites can be converted into the corresponding thiosulphates by treatment with a freshly prepared concentrated solution of ammonium pentasulphide, and then boiling off the excess of ammonia. Salt No. I. yields tabular crystals, having the formula $\text{NaKS}_2\text{O}_3 + 2 \text{H}_2\text{O}$; these are readily soluble in water, melt at 57°C , and have a specific gravity of 1.970 at 15° . Salt No. II. yields a similar thiosulphate also containing two molecules of water of crystallisation, but they melt at 62° , and have a specific gravity of 1.930 at 15° . When heated with ethyl bromide for some hours the former is converted into potassium ethyl thiosulphate, the latter into sodium ethyl thiosulphate, both crystalline compounds; the sodium and the potassium have been respectively replaced by the ethyl group, and since the ethyl group would in all probability attach itself to the sulphur atom united directly to the metal, the constitutional formulae for the two thiosulphates would be—

No. I.



No. II.



Both these thiosulphates treated with iodine are converted into potassium tetrathionate; salt No. II. should give sodium tetrathionate, but potassium iodide is formed in the reaction which decomposes the sodium tetrathionate with formation of the potassium salt as was proved by direct experiment. For a similar reason both the thiosulphates yield a potassium silver thiosulphate having the formula $\text{KAgs}_2\text{O}_3 + \text{NH}_3$ when treated with an ammoniacal solution of silver nitrate. This compound loses its ammonia at 100° , and leaves a brown powder; the aqueous solution readily decomposes with separation of silver sulphide. Sodium silver thiosulphate ($\text{NaAgS}_2\text{O}_3 + \text{H}_2\text{O}$) previously obtained by Lenz (Ann. Chem. Pharm. 40, 94) is also described.

The author draws the conclusions that the above facts point to the asymmetric constitution of the sulphites and thiosulphates.—C. A. K.

Nitrous Vitriol, and its Purification for the Manufacture of Aerated Waters. Pharm. Conference, Newcastle-on-Tyne. Pharm. J. 1889, 204.

At the *Pharmaceutical Conference* just recently held at Newcastle-on-Tyne, J. Pattinson read a paper upon the above subject. He had observed that when nitrous vitriol is employed to generate carbonic acid for charging ginger ale, the beverage obtained was more or less turbid. J. Pattinson found that the amount of nitrous compounds required to cause this turbidity was almost infinitesimal, 0.026 per cent. of N_2O_3 in the objectionable vitriol being sufficient to give rise to turbidity, whilst with the pure article a perfectly clear ginger ale resulted. Naylor pointed out that the pungency of the ginger essence, as well as that of the capsicin, were destroyed by the nitrous compound. T. H. Williams had also experienced the same troubles, but he added that, though ginger essence lost pungency and flavour, he had observed a similar effect on all kinds of aerated waters, and he found it was due to the nitrous fumes carried over in the carbonic acid gas. He had remedied this defect by passing the carbonic acid gas through a scrubber down which an alkaline solution of permanganate of potassium was allowed to trickle. The effect was all that could be desired. Pattinson proposed as a remedy to heat the vitriol with a little ammonium sulphate, which entirely destroys nitrous compounds. The presence or absence of these compounds can then be ascertained by adding a drop or two of permanganate to 20 cc. of the oil of vitriol, to which water is then added. If free from nitrous compounds, the pink colour is retained. The diphenylamine reaction might also be applied, and is still more delicate.—W. S.

PATENTS.

Improvements in obtaining Chlorine from Hydrochloric Acid or Chlorides, and in Regenerating Peroxide of Manganese which is used in the Process. A. Campbell, London, and W. Boyd, Glasgow. Eng. Pat. 10,187, July 13, 1888. 6d.

MANGANESE chloride (obtained by acting upon manganese peroxide with hydrochloric acid for the production of chlorine) is heated with sulphuric acid and a further quantity of manganese peroxide. The whole, or nearly the whole, of the chlorine is obtained in the free state, sulphate of manganese being formed at the same time. This latter is dissolved in water and treated with carbonate of ammonia. The precipitated carbonate of manganese is separated, and converted into peroxide by any suitable means; the sulphate of ammonia is recovered by concentration and fishing in the usual way. Three modifications of the process are described.—A. R. D.

Improvements in the Manufacture of Permanganates and Chlorates. G. H. Bolton, J. R. Wyble, and H. Auer, Widnes. Eng. Pat. 10,193, July 13, 1888. 6d.

A SOLUTION of manganate of soda which has been treated with chlorine, as described in Eng. Pat. 8217 of 1887 (this Journal, 1888, 437), and which contains sodium chloride and chlorate, as well as permanganate, is evaporated to about 90° Tw. During this process about two-thirds of the chloride present separates out and is removed by fishing. Potassium chloride is now added, in quantity rather less than sufficient to convert all the sodium permanganate into the potassium salt, and the solution, standing at about 80° Tw., is allowed to cool. The bulk of the permanganate crystallises out, and a second crop of crystals may be obtained by a further concentration to 130°, with occasional removal of sodium chloride by fishing. The strong mother-liquor is freed from the remaining small quantity of manganese in solution by treatment with sulphuretted hydrogen or a soluble sulphide, after which it contains only the chlorate and chloride of sodium in about equivalent proportions. Further concentration to 100°–120° Tw., and fishing, removes a portion of the sodium chloride; the settled

liquor deposits on cooling chlorate of soda of great purity. If chlorate of potash be required, the necessary amount of potassium chloride should be previously added.—A. R. D.

Improvements in the Treatment of Sulphuretted Hydrogen to obtain Sulphur, and in Apparatus employed therein. J. Hargreaves, Ditton. Eng. Pat. 10,322, July 17, 1888. 6d.

SULPHURETTED HYDROGEN is intimately mixed with a volume of air just sufficient for the combustion of its hydrogen, and then caused to pass through a number of perforated blocks preferably built up of spiral-shaped pieces of earthenware or metal similar to those employed in the regenerator of the Hargreaves thermo-motor. These blocks are preferably placed one behind the other in a closed chamber, the intervening spaces being occupied by gauze-like material, to prevent the return of the flame. This end is better attained by making one of the blocks of metal, and keeping it cool by circulation of water. After passing the blocks the gaseous mixture is injected at a tangent into a tubular-shaped combustion chamber, made so long as to avoid sudden cooling before complete decomposition of the sulphuretted hydrogen. In order to recover as free sulphur any sulphur oxidised during the decomposition, the residual gases are passed through the solution of some suitable chloride, or they are scrubbed in any ordinary method for the recovery of the sulphur acids, and these, when concentrated, are injected into the hot end of the combustion chamber.—A. R. D.

Improvements in the Manufacture of Nitrate of Ammonia. C. N. Hake, London. Eng. Pat. 11,731, August 14, 1888. 4d.

It is proposed to produce solid nitrate of ammonium by bringing together nitric acid vapour and ammonia gas in a suitable mixing chamber, or by spraying liquid nitric acid into a chamber into which a current of ammonia gas is caused to flow. In the first case the nitrate is obtained as a fine powder; in the second as a supersaturated solution, solidifying on cooling. Care must be taken that the heat of combination be not allowed to raise the temperature in the mixing chamber above 120° C.—A. R. D.

Improvements in the Manufacture of Chloride of Aluminium with other Chlorides. W. White, Cheshunt. Eng. Pat. 12,543, August 31, 1888. 4d.

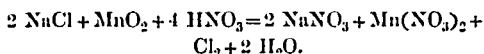
SODA or potash alum is dried, powdered, and intimately mixed with enough sodium or potassium chloride to convert the aluminium of the alum into aluminium chloride and to form with the same a double chloride. When the mixture is fused at a red heat the double chloride sublimes, leaving potassium or sodium sulphate behind in the retort or crucible.—A. R. D.

Improvements in the Manufacture of Soda Alum. E. Augé, Montpellier, France. Eng. Pat. 6443, April 15, 1889. 4d.

SOLUTIONS of sulphate of soda and sulphate of alumina when mixed together in the proper proportions cannot well be evaporated in the ordinary way to the density necessary for crystallisation without employing a temperature of at least 70°–80° C., and the soda alum solution when heated to this degree is rendered incapable of crystallising. The inventor obviates this difficulty by conducting the evaporation under reduced pressure, and obtains the required specific gravity (1.38 to 1.46) at a temperature of about 48° C.—in no case exceeding 60° C.—A. R. D.

Improvements in the Process of Manufacturing Caustic Soda or Caustic Potash. W. P. Thompson, Liverpool. From J. A. Bradburn, Syracuse, U.S.A. Eng. Pat. 6710, April 20, 1889. 4d.

To two molecules of sodium chloride are added four of nitric acid and one of peroxide of manganese. The mixture is heated by steam in a stoneware retort, the following reaction taking place:—



The chlorine thus produced is used in the ordinary way for making bleaching powder. The manganese in the residual liquors is precipitated with caustic soda, and then oxidised by a current of air, which renders it again available for use in the first stage of the process. The nitrate of soda left in solution is recovered by evaporation, mixed with ferric oxide, and heated in a current of air. The mixture of nitrous gases is passed over some oxidising manganese compound and treated for the recovery of the nitric acid. The mass remaining in the furnace yields on lixiviation a solution of caustic soda. The remaining ferric oxide can be used over again. Instead of manganese peroxide a manganate or permanganate may be used in the first operation. The process is equally applicable for the production of caustic potash.—A. R. D.

A New or Improved Process for making Metallic Sulphates in Solution. H. E. Newton, London. From L. O'Brien, Sydney, New South Wales. Eng. Pat. 8045, May 14, 1889. 4d.

THE ores or minerals to be dissolved are placed in water in a vat provided at the bottom with a chamber into which are pumped under pressure sulphurous acid and steam. The mingled gases escape from this chamber by slits in its sides, and bubbling up through the contents of the vat, effect the "sulphating" of the ore therein. The boiling temperature, requisite for speedy solution is maintained by the steam with which the sulphurous acid gas is mixed. In certain cases the action may be accelerated by the help of an oxidising agent such as nitrous acid or oxide of manganese. —A. R. D.

VIII.—GLASS, POTTERY, AND EARTHENWARE.

The Solubility of Glass Bottles. R. Reynolds. Pharm. Conference, Newcastle-on-Tyne.

REUTER (*Pharm. J.*, Aug. 31, 1889) showed that chloral hydrate (cryst.) which had been kept some time in a blue glass bottle, acquired a blue tint, the colour being proved to be due to pigment derived from the glass of the bottle. Glass is known to be affected by alkaline solutions, and the glass windows of stables even become gradually attacked by the ammoniacal emanations from decomposed and decomposing urine. It was also pointed out that glass that has lain in mud becomes disintegrated, and hydrocyanic acid kept in white flint glass bottles becomes decomposed, owing apparently to the action of the glass. As a matter of personal experience, Reynolds mentioned that a solution of 1 in 10,000 of hydrochloric acid in water, which when freshly made gave a distinct reaction with congo-paper, failed to do so after being kept a day in a flint glass bottle. This result he was inclined to refer to the chemical interference of the bottle with its contents. The observation was confirmed in a similar one by Thresh, who, in testing the action of acid fumes in the

smoke of a neighbourhood on vegetation, found that whilst the rain collected directly on litmus papers was acid, that collected first in glass bottles showed either a neutral or an alkaline reaction. He tried all kinds of bottles, with a similar result. Some of the rain water was then collected in platinum dishes, and the water collected in bottles gave him 5 grains per gallon more solid residue than that collected directly in platinum. This residue was chiefly siliceous, pointing to the fact that a decomposition of the silicates of soda or lime, or both, had probably taken place.

Linford stated that hydrochloric acid acted very considerably on bottles, but sulphuric acid did not. The action of hydrochloric acid, however, soon ceased, and the bottle was no longer acted upon. Martindale had observed that *osmic acid* very much affects white glass bottles. The English-made bottles were chiefly affected. Nearly all foreign glass bottles stood the action of the acid exceedingly well. Where the grinding of the stopper occurred the action was only slightly noticeable. This result was considered to be due to the fact that English bottle glass contains more lead than the foreign glass. Bottles made of glass containing no lead would be found to stand the osmic acid very well. Weddell confirmed the statement that English bottle glass contains a considerable percentage of lead, whilst the French and German glasses are free from that metal.—W. S.

Mexican Pottery Glaze.

IN the current number of *Harper's Monthly Magazine* is an interesting announcement of a discovery of a band of Mexican Indians who manufacture a pottery glaze, which, in an introduction to the article, the expert in potteries, W. C. Prime, compares to the wondrous old glazes of Gubbio and the Florentine potters. "The fact stands," he says, "that to-day in San Felipe unskilled Indians are producing and selling for daily use in Mexico wares decorated with a lustre incomparably finer than any metallic lustre produced in Europe, and which, if potters can by experiment learn so to use it that different colours can be produced as desired, will practically restore to modern uses the lost Sarracen and Italian lustres."

As much of the secret as the San Felipe Indians possessed is out, for at some risk Miss Y. H. Addis has penetrated to where the pottery was made, and has secured the entire process of firing, &c., adopted by the makers of these beautiful wares. She thus describes it: "The vessels once moulded, are fired, and when thoroughly cold are glazed with a mixture of (1) oxide of lead; (2) broken glass, which they buy from the refuse of shops; (3) 'peacock copper;' and (4) a very fine sand found near San Felipe. It was impossible to obtain even an approximately accurate idea of the proportions employed, because the potters have so long worked by the rule of thumb that they are really incapable of describing the quantities. Therefore, this point would have to be determined by examination during their practical working. After application of the glaze comes a second firing—the one in progress at the time of the visit. The ware emerged from the kiln at this stage dull and clouded, of a thick muddy brown or greenish colour. As rapidly as possible the potter buried the pieces in a great heap of stable manure, and after from one to hours they were disinterred, transformed by the ammoniacal fumes to a lustrous brilliance, with the shifting, varying hues that gleam on the breast of a peacock. The secret, if secret there be, seems to lie in two of the elements of the glaze, in combination with the ammonia treatment, for I am sceptical as to the potter's assertion that the effect is due to peculiar properties in the clay of San Felipe."—*Engineering and Mining Journal*.

PATENT.

Improvements in the Manufacture of Red Glass. H. J. Haddan, London. From the Josephine Glass Works of Count Schaffgotsch, Schreiberhau, Germany. Eng. Pat. 11,016, July 30, 1888. *6d.*

The following mixture is prepared and melted in an open pot:—

	Parts.
Fine sand	2,000
Minium	400
Potashes	600
Lime	100
Phosphate of lime	20
Tartar (bitartrate of potash)	20
Borax	20
Red oxide of copper	9
Binoxide of tin (putty powder)	13

This gives at one melting a homogeneous glass of a transparent red colour, from which articles may be made direct without flashing, and without the necessity for a second heating to bring out the colour.—A. R. D.

IX.—BUILDING MATERIALS, CLAYS, MORTARS, AND CEMENTS.

Natural Cements. A. Cluss. Eng. and Min. J. 47, 546.

LIMESTONE, with intimate fine-grained admixtures of silica, alumina, magnesia, &c., in quantities of 35 to 60 per cent., are called cement stones. The high percentage of silicates causes a separation of the alumina from the silica during calcination at a red heat just sufficient to expel the carbonic acid. Oxides of iron and chloride of potash are generally present in small quantities. The burned lumps must be pulverised before they will combine with water, when they form hydrated silicate of lime, while the alumina remains practically inert. These cements set quickly, but harden slowly under water without shrinking, and attain gradually great strength, with well developed adhesive force. They bear doses of sand of double their own volume or over. From 30 to 40 per cent. of water must be added. Many American cements of this class contain large percentages of carbonate of magnesia. The tests of strength, in pure state as well as mixed with sand, compare well with the cements which do not contain these combinations. De Smeth found for Virginia cements in the pure state, after 30 days' exposure, 170–250 pounds tensile strength per square inch, which increases in 11 months to 316–381 pounds. Gilmore states the adhesion of Rosendale cement to the front bricks, after 28 days, when pure, to be 30 pounds, and mixed with one and two parts of sand, 16 and 12 pounds.

Clarke reports the tensile strength of Rosendale cements, pure:—

	Pounds.
After 1 and 12 months, as.....	115 to 290
And mixed 1:1 after 1 and 12 months.....	116 to 250
1:2 after 1 and 12 months.....	60 to 180
1:3 after 1 and 12 months.....	35 to 121
One cubic foot of Rosendale cement weighs.....	49 to 50

The proportion of tensile to compressive strength averages probably after one month 1:4, and rises probably, after two years, to about 1:6.—A. J. S.

Proceedings of the Austrian Society of Chemical Industry. Meeting of June 1, 1889. Chem. Zeit. 13, 781.

K. ZULKOWSKI criticised the work of Knapp on blast-furnace slag and Portland cement, in which the latter referred to the similar behaviour of the two materials when treated with dilute hydrochloric acid, ammonium chloride, magnesium chloride, or sugar, which dissolve lime freely from

them without bringing the corresponding amount of silica into solution. From this fact Knapp concluded that the lime so extracted is not in either material combined with silica. Zulkowski differs from this view. Twenty-six years ago he found that lime could be easily extracted, even by weak reagents, from basic alumino-silicate of lime which had been formed by heat, only inconsiderable amounts of silica dissolving at the same time. Again, Mylius and Foerster have shown that alkalis are dissolved out of glass by pure water, and that very little silica accompanies the alkali so dissolved. Water, further, dissolves lime from hydraulic lime, without it being thereby proved that the lime was present in the free state, a fact which can only be proved by a synthetical experiment. Zulkowski maintains the correctness of his theory, which is as follows:—

- (1.) That, in the burning of natural or artificial hydraulic lime, the clay is decomposed, with the formation of a basic silicate.
- (2.) When this basic silicate is fused, as in the case of blast-furnace slag, the whole of the lime becomes combined and the hydraulic property is lost.
- (3.) When the mixture is heated beyond fusion, there results a mixture of finely-divided lime and a fused or cindery basic silicate, which, in presence of water, can again combine with the excess of lime.—E. B.

X.—METALLURGY.

Treatment of Quicksilver Ore Fines at Almaden, Spain. Eng. and Min. J. 47, 544–545.

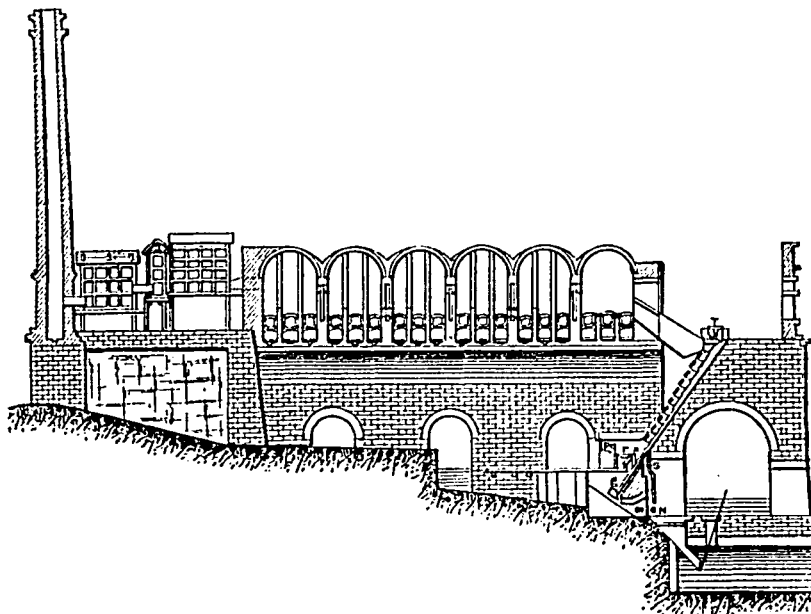
CHANNEL furnaces are employed for the treatment of fines rich in quicksilver at Almaden. The size of the fines for these furnaces must not exceed 10 cubic centimetres. The furnaces mentioned have the great advantage of being continuous in their operation, to such an extent that in seven months not the slightest interruption occurred.

The hearth is an inclined plane constructed of fire-brick, 2.50 metres in width by about 8 metres in length, the grade of which is equal to, or a little steeper than the natural slope of the fines. Every 40 centimetres this floor has a rise of 2 centimetres, as can be seen in the illustration. It is divided by partitions, formed of fire-brick, 30 centimetres high, into 12 channels, 12 centimetres wide, in which the ore runs. Transversely in the channels, and supported on their partitions, there are placed bricks, called "taws" ("stoppers"), which are above the floor of the hearth 4 centimetres, this being the thickness of ore in each channel. Arranged in the same way there are others, called "petacos" ("trunks"), situated immediately at the bottom of the rises and at the same distance from the hearth as the "stoppers," which serve to break up the fine ore on them so as not to present invariably the same surface to the action of the heat. On the lower part of the hearth there is another inclined plane, M, perpendicular to it. This is also divided into 13 channels, but there are no "stoppers" nor "trunks" on it. The ore, already calcined on the first plane, travels to the second, at the end of which, M, a boy is placed, whose duty is to make it run over the hearth. A little above the second plane, and facing the first, but separated from it by the bridge *x*, is placed the fire-place, the grate of which is divided into two equal parts by a partition, which rises to the arched roof of the fire-place. The fire-grate is formed of two systems of bars, *p*, *q* and *r*, *s*, the first placed lengthwise and irregular in height, and the second crosswise. The air for combustion passes in by the ash-pit to the fire-place, having traversed certain passages *c*, *f*, *g*, where its temperature has been raised by the heat taken from the walls of the furnace. By means of the hopper "T" the channels are filled with ore, and when it has been sufficiently exposed to the heating and reducing action of the gases, the boy placed at M withdraws

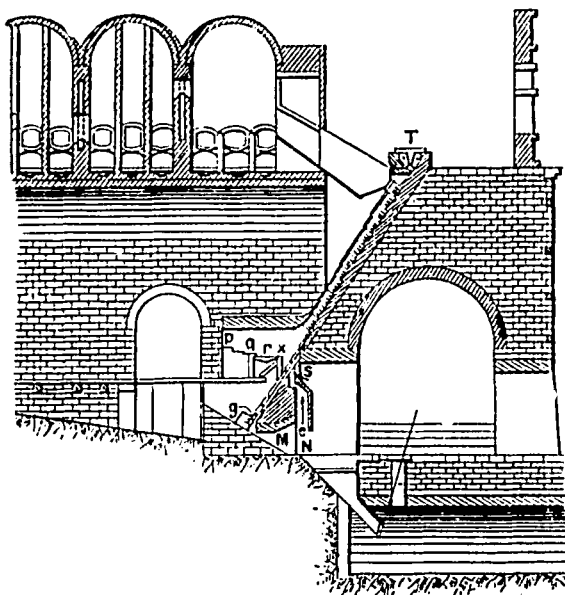
a portion of the ore placed on the lower edge of the second inclined plane. The ore remains subjected to the action of the heat about four hours. The calcined fines are ejected by a hopper to the passage G, whence they are taken out in

cars. They do not now contain more than two to four hundredths per cent. of quicksilver. The condenser is formed by two series of chambers, the first of which communicates with the furnace by a throat in the shape of a

FURNACE FOR CINNABAR FINES AT ALMADEN.



ENLARGED SECTION OF THE ABOVE.



truncated pyramid of iron plate protected with brick. The six chambers which constitute this first series are of brick, with thin walls, and are divided into partitions, which have openings arranged for the passage of the fumes, so as to run in double zigzag. The bottom of each division is formed of two inclined planes, whose intersection has a small incline towards a channel common to all the chambers in which is collected the quicksilver condensed in them. The bottoms are of iron plate in the three chambers nearest to the furnace, and of slate in those furthest away, this arrangement being in consequence of the iron being attacked

by the quicksilver in the latter. The last part of the condenser is formed of wooden chambers with glass, divided into four parts by vertical partitions. These chambers are arranged so that the air can circulate below and around the sides, and allow of observation in case of any filtration of quicksilver through the bottoms.

Coal was first employed as fuel, but coke is now found to give better results in regard to the smaller quantity of soot which is produced.—A. J. S.

The Platinum Metals. Eng. and Min. J. 47, 586—583.

Platinum.—Usually found in the form of small rounded and flattened grains. Platinum alloyed with other members of this group is found in small quantities in the beach sands and gold placers of the Pacific coast, and also associated with placer gold in Virginia, North Carolina, Georgia, and other southern States. In 1887 a nugget, weighing 104·4 grains, was described as found near Plattsburg, N.Y. Sperrylite, a mineral containing platinum and arsenic, was discovered in the autumn of 1888, at the Vermillion mine, near Sudbury, Ontario, Canada. It was found to contain upwards of 7·52 per cent. of platinum. The greater part of the platinum that finds its way into commerce is derived from placer deposits in the districts of Soroblogodar and Nishni-Fagikk, in the Ural Mountains. Its refining is largely accomplished by the house of Johnson and Matthey, of London, where Deville's process is used, and the French corporation in Paris, formerly known as Quenessen, Le Brun, and Dessuontis. The method of mining is analogous to that of placer gold, with the exception that the apparatus for saving platinum depends upon gravity alone, and not upon gravity and amalgamation. From the material thus accumulated platinum is derived in the following method: The platinum is precipitated as a platino-chloride of either potassium or ammonium in the nitro-hydrochloric acid solution of the crude metal. This substance washed with a little cold water, dried and heated to redness, or reduced in a current of hydrogen, leaves metallic platinum in a spongy state. The spongy platinum is made into a thin uniform paste with water, introduced into a slightly curved mould of brass, and subjected to a gradual pressure, by which the water is squeezed out, and the mass rendered at length sufficiently solid to bear handling. It is then dried, very carefully heated to whiteness, and hammered or subjected to powerful pressure, when the platinum will be in a state to bear forging into a bar. Deville and Debray's method of refining consists in heating the crude metal in a crucible of lime by the oxy-hydrogen flame. All the impurities are separated in this way.

Palladium.—This metal is separated from the other members of the group by neutralising the acid solution, from which the platinum has been precipitated with sodium carbonate, and mixing with a solution of mercuric cyanide. Palladium cyanide separates out, and on being washed, dried, and heated to redness, yields metallic palladium in a spongy state. There has recently been described an alloy consisting of from 60 to 75 parts of palladium, 15 to 25 parts of copper, and one to five parts of iron, and its use recommended as suitable for the manufacture of watches owing to its non-magnetisable qualities. Other palladium alloys were described containing varying proportions of silver, gold, platinum, and nickel.

Rhodium.—From time to time it has been reported that rhodium occurs in small quantity in the amygdaloid rocks of Keweenaw Point, Lake Superior; but it is believed that its presence has not been authoritatively established. This metal remains in the solution after the platinum and palladium have been removed, and is separated in the following manner: The solution is mixed with hydrochloric acid and evaporated to dryness, when the residue is treated with alcohol of 0·837 density, which dissolves everything except the double chloride of rhodium and sodium. This is well washed with alcohol, dried, heated to whiteness, and then boiled with water, by means of which the sodium chloride is dissolved out, and metallic rhodium remains. It is a white coherent, spongy mass, less fusible and less capable of being welded than platinum. A small quantity of it in steel is said to impart most valuable qualities.

Iridium.—Its principal source is the mineral iridosmine, which, in America, is found chiefly in the placer washings of California and Oregon, where it accumulates with the gold on account of its weight, and is easily recovered and saved. When the crude platinum is dissolved in nitro-hydrochloric acid, a small quantity of a grey scaly metallic substance usually remains behind, having altogether resisted the action of the acid. This is the native alloy of iridium and osmium. It has been found in Cincinnati that by heating iridium or any of its ores in a sand crucible to a

white heat, and then adding phosphorus, the latter rapidly combined with the iridium, and in a few seconds the contents of the crucible were in a perfect state of fusion, capable of being poured and cast into an ingot. This material proved to be as hard as iridium, and seemed to have all the properties of the metal itself. The standard meter of Paris was made of an alloy consisting of 90 parts platinum and 10 parts iridium. Iridium has been used by the American Iridium Company, of Cincinnati, for the so-called diamond points upon gold pens, for the points of stylographic pens, for the knife edges of fine balances, for the tips of rubber turnery tools, for wire draw-plates in electric lighting, for the electric contact points of telegraphic apparatus, and, indeed, for any purposes where an exceedingly hard metal is required. Deposited electrolytically on base metals, it has been found to be exceedingly tough and susceptible of a high polish.

Ruthenium.—To obtain ruthenium, scaly osmiridium is heated to bright redness in a porcelain tube, through which a current of air (freed from carbonic acid and from organic matter) is drawn by means of an aspirator. The osmium and ruthenium are thereby oxidised, the former being carried forward as tetroxide and condensed in solution of potassium hydroxide, while the ruthenium oxide remains behind, together with iridium, and by fusing this residue with potassium hydroxide, treating the mass with water, and leaving the liquid in a corked bottle for about two hours to clarify, a solution of potassium ruthenate is obtained, which, when neutralised with nitric acid, deposits velvet-black ruthenium sesquioxide, and this, when washed, dried, and ignited in hydrogen, yields the metal.

Osmium.—When ruthenium and osmium are oxidised, the latter as tetroxide is passed in the form of a vapour into a solution of potassium hydroxide. This solution is mixed with an excess of hydrochloric acid and digested with mercury in a well closed bottle at 40° C. The osmium is then reduced by the mercury and an amalgam is formed, which, when distilled in a stream of hydrogen till all the mercury and calomel are expelled, leaves the metallic osmium in the form of a powder. Deville and Debray, by igniting precipitated osmium sulphide in a crucible of gas coke, at the melting heat of nickel, obtained it in bluish-black, easily divisible lumps.—A. J. S.

Note on the Influence of Temperature on the Mechanical Properties of Iron and Steel. A. Le Chatelier. Compt. Rend. 109, 58—61.

The author has observed a noticeable change in the properties of iron and steel at a temperature above 80° and again a further one at about 240°. The change is permanent. There is an increase of elasticity and a notable reduction in the elongation. Thus three iron wires 2 metres long were loaded with 30 kilos. per square mm. at 15°, which gives an elongation of 9 per cent. No. 1 was now heated to 74°, No. 2 to 92°, No. 3 to 190°, each for 10 minutes. After cooling the following results were obtained:—

—	Limit of Elasticity.	Load.	Elongation.
No. 1.....(71°)	Kilos. 33·5	Kilos. 38·2	Per Cent. 21
No. 2.....(91°)	30·0	37·4	2
No. 3.....(190°)	40·0	41·0	4

Ordinary puddled iron drawn into thin wires was experimented upon and gave very irregular results, owing to minute particles of slag contained in the metal. The irons and steels used for the experiments referred to were of great purity. They contained 0·35 to 0·40 per cent. of manganese, and whilst the irons contained 0·05 per cent. of carbon, the steels contained 0·80 per cent. of that element.

—A. R.

Distilled Lead. R. Roesing. Chem. Zeit. 695—696.

THE lead ores from Upper Silesia are specially suitable for the manufacture of pure lead, as they contain no bismuth or copper and only minute quantities of gold, antimony, arsenic, and nickel. Silver, zinc, and iron are present, but can be removed without difficulty. A sample of the lead produced by the distillation process at the Kgl. Friedrichshütte specially for chemical purposes, and from these ores, contained: Copper, 0.0007; antimony, 0.0006; arsenic, 0.0013; iron, 0.0010; traces of zinc, silver, bismuth, and nickel, lead (by difference), 99.9964 per cent.—E. E. B.

On the Nature of Cobalt and Nickel. Fleitmann. Chem. Zeit. 13, 757.

THE author has repeated the experiments of Krüss and Schmidt with different kinds of commercial nickel and cobalt oxides, using as much as 50 grms., without obtaining a weighable quantity of the new metal. He has unsuccessfully endeavoured to obtain a sample from Krüss and consequently considers "Gnomium" as a phantasy. His own researches lead to the following conclusions:—(1.) If a new metal really exists as a general constituent of nickel and cobalt it cannot be present to the amount of 2 per cent. (2.) That this small quantity can have no real influence on the malleability of nickel, which in its usual purity is as weldable and malleable as the softest iron. (3.) That the patented process of Krüss is worthless, while it is not new and is partly impracticable.

If commercial nickel and cobalt in the form of hydroxides be treated with large quantities of caustic soda one obtains varying amounts of impurities which are usually present, together with small quantities of nickelous and cobaltous oxides. These impurities vary according to the source of the metal, but may consist of mixtures of lead oxide, zinc oxide, arsenious acid, manganic acid, molybdic acid, silica, alumina, cerium oxide, chromic acid, &c. The simultaneously dissolved nickel and cobalt oxides do not, as a rule, represent more than one-twentieth per cent. of the oxides taken. If they are separated from the remaining impurities which are dissolved by the alkali, and converted into nickelous and cobaltous oxides, they are found to be insoluble in caustic soda, by which it appears that their original solubility is due to the presence of some of the impurities, such as arsenious acid, silica, and alumina. If the original alkaline extract is treated with hydrochloric acid until the precipitate formed is redissolved, and then with ammonium carbonate, a complex mixture of different oxides and acids is obtained, which might confuse and mislead anyone not acquainted with such a mixture. It seems probable that Krüss and Schmidt had to deal with a similar mixture. The precaution taken by them to precipitate at a temperature below 10° C. strengthens Fleitmann's conclusion, since on warming precipitates are frequently formed, due to the manganic acid and the dissolved lead oxide being converted into manganic oxide and lead peroxide.—A. W.

Relative Merits of Different Lead-Tin Alloys for Soldering. J. Rothe. Mitth. Königl. techn. Versuchs. 1889, 86—87.

TWO alloys were prepared from pure lead and Banca tin. A consisted of 10 per cent. of lead and 90 of tin, and melted at 210° C.; B was composed of 30 per cent. of lead and 70 of tin, and fused at 188° C. A was harder and less malleable than B, but behaved like it when subjected to bending tests; it gave the "cry" of tin, while in the case of B this property could scarcely be perceived.

Mechanical tests showed that A was as efficient a solder as B. Experiments with cast plates of the alloys exposed to the action of acetic acid indicated that at first B was more strongly attacked, and yielded more lead absolutely and relatively to the acid; after 24 hours tin was still freely dissolved from both alloys, but the amount of lead removed decreased considerably and became approximately constant and equal for both. It therefore appears that the lead

dissolved at the outset is reprecipitated by the more electro-positive metal, and is protected fairly completely by it subsequently.—B. B.

Servian Quicksilver.

THE attention called to the new Servian quicksilver industry by the floating of the Avala Quicksilver Mines Company (Limited) in London recently, gave a recent visit made by us to the Servian section of the Paris Exhibition a more than passing interest. The Avala mines are situated fifteen miles south of Belgrade, and about two from the main railway line which connects that city with Paris and Constantinople. The mines are to be found at the foot of the mountain from which it takes its name, itself deriving its present name from an ancient castle, now in ruins, dating its origin to a century or two B.C., and with which many interesting legends are connected. The mines were discovered in 1883, the intervening five years having been spent in experimental working.

At the present time there are five veins of quartz in which the quicksilver is found, the largest of these being about 60 yards in width and 700 yards in length, the first mine having reached a depth of 100 yards. Nor is this all, for in the same mineral is found a considerable amount of sulphate of nickel, besides a certain quantity of the natural metal. One hundred and fifty thousand tons of mineral have been raised, giving an average yield of 27 lb. of quicksilver per ton, the whole of which is said to equal the best qualities found in the cinnabar mines of Almaden, Spain, and the New Almaden mines of California. Sixty miners and an engineer are constantly at work, the operations going on both day and night, now showing a daily raising of 15 tons. Four kilns and 10 retorts, with a powerful steam-engine, seem at present to constitute the principal working plant of the mine. At Avala the extraction of the metal from its ore is effected by oxidation and reduction, a process which appears to differ from that in use at Almaden, where great waste occurs, owing to an imperfect method of condensation, while at the Servian mines a loss of 6.6 per cent. only is sustained. Twenty-five bottles of quicksilver are produced weekly, each bottle containing 70 lb. net weight of the metal. These bottles have a similar form and style to those used in England for the nitrous oxide gas. Five hundred of them recently found a ready sale on the London market, while 200 have been despatched to China. At present, however, the company seems disposed to limit its sales to Vienna, where prices range somewhat higher than elsewhere, besides the great economy obtained in the matter of carriage, as compared with London, &c. It is not to be supposed that the company in question has been without competitors, but these, it would seem, from some unexplained reason, have not been successful. The principal one was the Kipanj syndicate. A former English concern, too, which worked the copper mines at Meidenpeck some years ago, came to grief; this was the first, and up to now the only, attempt by capitalists from this country to develop the mineral resources of Servia. We have searched in the United States and Spanish courts, consulted the officials, and perused the catalogues, but can hear of no other quicksilver exhibit. This is a matter of regret, as it would undoubtedly have been interesting to give some information from other sources.—*Chemist and Druggist.*

PATENTS.

Improvements in the Manufacture of Metallic Alloys. G. A. Dick, London. Eng. Pat. 2484, May 25, 1882. (Second Edition.) 6d.

See this Journal, 1883, 285.—H. S. P.

Improvements in or Relating to the Treatment of Ores for Facilitating the Extraction and Recovery therefrom of Gold and Silver, and in Apparatus therefor. W. P. Thompson, Liverpool. From H. F. Julian, Johannesburg, South Africa. Eng. Pat. 8332, June 7, 1888. 8d.

THE finely pulverised ore is rotated in an air-tight barrel with water and chlorine, under a pressure of 60 to 80 lb. per square inch, produced by "air, steam, or other fluid."

Mercury is then introduced into the same or another barrel with the mud, and the whole again revolved under pressure. The contents are then run over copper plates to retain the amalgam, and, finally, the tailings are washed through a series of electrolytic cells in which the cathodes are mercury and the anodes carbon or other suitable material. These cells are arranged at different levels and overflow into each other. They are said to remove the last traces of gold, and catch any floured mercury or amalgam. Drawings are given.—A. W.

Improvements in Carburising and Coating Iron and Steel in the Form of Sheets, Hoops, Wrought or Cast Plates, Bars, or Manufactured Articles. J. Summerhill, St. Georges. Eng. Pat. 9848, July 6, 1888. 6d.

THE steel or iron articles are heated in an annealing furnace or muffle to as high a temperature as it is possible to attain without occasioning oxidation of the metal, and are then withdrawn and dipped into a bath of carbonaceous material, such as tar, pitch, or oil. The metal thereby becomes carburised and likewise coated with an adherent carbonaceous protective coating, which has a glazed appearance similar to Japan black when dried in the ordinary atmosphere, or a dull surface when artificially dried by heat. The most suitable bath is composed of approximately pure tar, containing 1 per cent. of Val de Travers rock. The time of immersion varies with the thickness of the material, and also with the desirability of carburisation with or without the production of the carbonaceous coating.

—A. W.

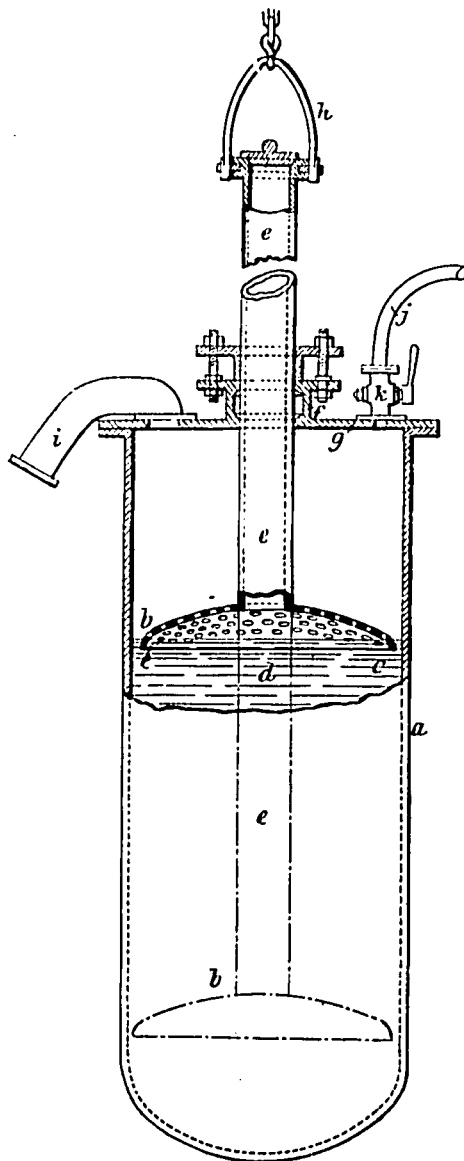
Improvements in Certain Metallic Alloys. E. L'Homme, Paris, France. Eng. Pat. 2722, July 19, 1888. 6d.

THE patent relates to the production of bronze or brass that possesses great hardness, toughness, and tensile strength, and which is capable of being rolled or otherwise worked in either the hot or cold state. The alloy consists of copper, iron, manganese, zinc, nickel, silicon, and carbon. The method of procedure is as follows:—In a suitable crucible or furnace are melted copper, nickel, ferro-manganese, ferro-silicon, and ferrocyanide of potassium, zinc is then introduced and thoroughly incorporated with other metals. The draught is then lowered and about 2 per cent. by weight of carbonate of soda is added, and a few minutes later the metal may be run off into moulds. The novelty consists in using ferrocyanide of potassium, whereby carbon is introduced into the alloy, and in the addition of a minute proportion of nickel to alloys of this class, which has the effect of lowering the melting point of the mass and increasing the homogeneity of the alloy. The proportions used are, to every 100 lb. of copper, 34—62 lb. of zinc, 4—6 lb. of ferro-manganese (containing 84 per cent. of manganese), 1—2 lb. of ferro-silicon (containing 14 per cent. of silicon), 2—4 oz. of nickel, and 10—30 oz. of ferrocyanide of potassium.—H. S. P.

Improvements in the Manufacture of Sodium and Potassium and the Apparatus therefor. W. G. Forster, London. Eng. Pat. 10,785, July 25, 1888. 6d.

THE patent is for apparatus used in the following process for making potassium or sodium. A reducing agent, such as carbon or carbon and iron mixed, is introduced gradually and from time to time, and preferably in a heated condition, into a bath of fused incandescent alkali, such alkali being either a carbonate or hydrate, or wholly or partially deprived of its hydrogen and oxygen by the use of finely-divided iron while in a fused condition. The invention consists of a contrivance for mixing the carbon with the fused alkali and preventing it from floating at the top of the bath. The alkali is fused in an upright cylindrical iron vessel *a*, fitted with a cover *g*. Into this vessel is placed a perforated iron disc *b*, of nearly the same diameter as the vessel. The disc is curved downwards at its rim and is attached to a hollow handle *e*, longer than the vessel. This handle passes through a stuffing box *f*, in centre of the cover, and by attaching it to

a chain and pulley or to a lever the disc may be moved up and down in the vessel, thus submerging the carbon and agitating the charge of alkali and carbon. The vapours and



gases generated in the process pass away by a pipe *i*, let into the cover of the vessel. The hollow handle of the disc is fitted with a loose cover and serves as a pipe for introducing the charge of alkali and the additions of carbon. It may also be used for discharging the waste alkali at the end of the reduction. For this purpose the loose cover of the hollow handle is removed and a short bend attached. The escape pipe for the vapours is then closed and the disc lowered nearly to the bottom of the bath. The confined gases then force the liquid residue up through the handle and through the bend into a suitable receptacle. Or, if preferable, coal gas may be forced into the vessel through inlet-pipe *j*, under sufficient pressure to expel the liquid residue. *k* is the stop-cock for pipe *j*. One of the chief advantages of the invention is that much larger vessels may be employed than those usually used in the manufacture of potassium and sodium.

The claims are:—1. In the manufacture of sodium and potassium, keeping the carbon submerged in the fused incandescent alkali by mechanical means. 2. The use of

apparatus, as described and shown, for the above purpose and for agitating the carbon with the heated liquid alkali, and allowing escape for the vapours produced in the process. 3. The improved apparatus.—H. S. P.

Improvements in Metallic Alloys. P. M. Parsons, Blackheath. Eng. Pat. 11,512, August 9, 1888. 4d.

THE patent relates to alloys of copper and zinc, or copper and tin, or copper, zinc, and tin together with steel, iron, and manganese, which last may be added as ferromanganese or spiegeleisen, as described in Eng. Pat. 482 of 1876. The patentee now adds aluminium to such alloys, whereby their strength is increased, they are rendered more fluid when molten, and oxidation is diminished so that from such alloys very sound and sharp castings can be made. He finds that one-eighth of one per cent. of aluminium has an appreciable influence on the character of the alloy, and this amount may be increased with advantage to about two to three per cent. A still larger quantity of aluminium may be used if a very hard refractory metal is wanted, but at the same time the alloy becomes more brittle. These qualities may both be modified by reducing the tin or the zinc as the aluminium is increased. The aluminium may be added in any convenient way. For instance, after fusing all the other ingredients, pieces of aluminium may be introduced into the fused mass by means of a pair of tongs or other suitable appliance, and then, by stirring, the aluminium becomes melted and combines with the other metals.

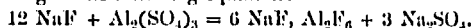
—H. S. P.

A New or Improved Process for Extracting Gold, Silver, and other Metals from Refractory Ores, Sands, and Residues. E. de Rottermund, Bladow, Russia. Eng. Pat. 12,074, August 21, 1888. 8d.

THE process consists of five operations:—(1.) Roasting; (2.) The extraction of the secondary metals; (3.) The removal of the protoxide salts; (4.) The extraction of the fine gold; and (5.) The final washing of the residue to collect the coarse gold. The roasting is carried on by preference in a Muekkell furnace, and is modified according to the nature of the ore. The roasted material is then leached with warm dilute sulphuric or muriatic acid, which “transforms the protoxides and salts at minimum of oxidation into oxides and salts at maximum of oxidation.” The liquor obtained is treated with scrap iron by which the copper, silver, &c. are precipitated. The residue is submitted to the action of nascent chlorine by running in a solution of chloride of lime simultaneously with dilute hydrochloric acid, whereby a solution of chloride of gold is obtained which is at once run off to prevent further contact with the ore, “which would tend to reduce again to the metallic state the gold contained in the said solution.” The solution also contains some silver, both which and the gold are extracted by ordinary methods. Any coarse gold remaining in the residues may be separated by mechanical means. The feature of this process is stated to be that only the most finely-distributed gold or that which is unobtainable by mechanical means is extracted by the chlorine, after which the coarse gold is recovered.—A. W.

Improvements in the Manufacture of Artificial Cryolite and similar Compounds. C. Netto, London. From C. Winkler, Freiberg, Germany. Eng. Pat. 12,135, August 22, 1888. 6d.

SLAG from the manufacture of aluminium from cryolite is melted with dehydrated sulphate of alumina in the proportion of 100 parts of the former to 32 parts of the latter, whereby the fluoride of sodium is decomposed with the formation of the double fluoride of sodium and aluminium according to the following equation:—



The sulphate of sodium is separated by lixiviation. Artificial cryolite can thus also be prepared from sodium or potassium fluoride in the first instance.—A. W.

A Method of Utilising Flux Skimmings from Galvanising Works. H. J. Kirkman, Swansea. Eng. Pat. 17,623, December 3, 1888. 4d.

ON treating the flux skimmings from galvanising works with water, zinc chloride and ammonium chloride pass into solution, leaving an insoluble residue containing zinc. The zinc may be recovered from this residue by any of the known processes. To the aqueous solution obtained as above described, lime (in the form of milk of lime), gas liquor, magnesia, sodium hydrate, sodium carbonate, or ammonia, is added in sufficient quantity. The precipitate of zinc hydrate or carbonate, as the case may be, thus formed, is separated from the solution, washed, dried, and, if need be, calcined. It may then be used as a pigment or the zinc may be recovered from it by the ordinary means. The solution, containing ammonium salts, may be treated by any of the known methods for the recovery of ammonia. Should any of the above mentioned substances, when added to the solution of the flux skimmings, decompose the ammonium salts and cause the evolution of ammonia, the solution may be treated in closed vessels and the evolved ammonia recovered by any of the known methods.—H. S. P.

Certain Improvements in the Amalgams, and Method of Applying the same, used in Amalgamation of Gold and Silver. M. Johnson, W. E. Field, and J. S. Beeman, Saint Kilda, Australia. Eng. Pat. 1602, January 29, 1889. 6d.

THE object of the invention is to prevent the “sickening” or “flouring” or “floating” of the mercury or amalgam used in extracting gold or silver, and in cases where the amalgam used has “sickened” or “floured,” to cause the particles to reunite or “quicken.” The invention consists in adding a small quantity of zinc (or cadmium or magnesium or other suitable metal, sodium and potassium excepted) to the mercury to be used, and after mixing the zinc amalgam thus formed with the auriferous or argentiferous ores under treatment, pouring upon the mixture dilute sulphuric acid or hydrochloric acid or an aqueous solution of caustic potash or other suitable alkali, or of chloride of ammonium or any other suitable salt, so as to generate hydrogen on or from the surface of the amalgam. The proper proportion of zinc to be used with a given quantity of mercury so as to form the zinc amalgam varies according to the nature of the ores or material under treatment, and can only be determined by experiment. For many classes of ores it has been found that 1 part of zinc to 90 of mercury forms a suitable amalgam. The process may also be carried out by amalgamating the gold or silver first and then adding the pieces of zinc and the acid, alkali, or suitable salt, to the mixture in the amalgamating pan or after removal from the pan. Or by mixing the mercury and the ore and the acid or alkali and allowing the whole to flow over, on or under plates or “riffles” which are covered with zinc amalgam. Or the ore and mercury together with the acid or alkali may be mixed and triturated in a pan of zinc or covered with zinc.—H. S. P.

Improvements relating to the Galvanising of Metals and to Apparatus therefor. T. Midgley, Beaver Falls, U.S.A., and W. B. Nye, Boston, U.S.A. Eng. Pat. 8255, May 17, 1889. 6d.

THE material to be coated with zinc such as iron wire, sheets, pipes, &c., is made to pass, after having been cleaned with acid, through a bath of molten lead and upwards through a small body of melted zinc floating on the surface of the lead and confined within a small area by suitable moveable partitions. Drawings are given. The advantages claimed are: less oxidation of the zinc; less waste of zinc from the action of acids; a more flexible material, as the iron is not cold when it enters the zinc; and the prevention of the destruction of the pans usually employed for melting the zinc by confining the latter between partitions on the surface of the lead.—A. W.

XI.—ELECTRO-CHEMISTRY AND ELECTRO-METALLURGY.

PATENTS.

Improvements in Secondary and other Electric Batteries. H. Pieper, Liege, Belgium. Eng. Pat. 11,569, August 10, 1888. 6d.

To increase the capacity and efficiency of batteries they are subjected to a pressure considerably greater than that of the atmosphere, while in use; in the case of some gases, chlorine for example, sufficient pressure is maintained to liquefy them as they are formed by the electrolytic action. Sometimes the pressure is produced artificially by air, and at others by the gases themselves as they are given off in an hermetically sealed vessel.—B. T.

Improved Cell or Containing Vessel for Electric Batteries. H. G. C. Serrin, Paris, France. Eng. Pat. 12,163, August 23, 1888. 11d.

The new cell is formed by making any number of cavities of the required size in a piece of natural wood of suitable size, and using each of these cavities as one cell of a battery. The battery can be used for any of the ordinary purposes, and can also have any desired apparatus mounted upon it.—B. T.

An Improvement in Electrical Batteries. W. Clark, London. From L. Maiche, Paris, France. Eng. Pat. 13,215, September 12, 1888. 4d.

A SOLUTION of citrate of ammonia, of varying strength according to circumstances, is used as the improved exciting agent.—B. T.

Improvements in or relating to Galvanic Batteries. G. C. Dymond, Liverpool. From C. Gassner, Mainz, Germany. Eng. Pat. 18,754, December 22, 1888. 4d.

AN isolated coal electrode is placed in a vessel of zinc, and the space is filled up with an exciting compound made by dissolving one part of sal-ammoniac and one of chloride of zinc in two parts of water, and then adding four parts of plaster powder, thus forming a thin pulp-like mass, which hardens in a short time.—B. T.

Improvements in Galvanic or Primary Batteries. C. Maltby-Newton, Croydon. Eng. Pat. 7356, May 3, 1889. 6d.

THE positive plates are made up of oxide of lead and syrup of sugar or molasses, the oxide of lead used consisting preferably of a mixture of flake litharge and powdered litharge.

The syrup and oxide are mixed up to a stiff paste, spread over each side of a perforated iron, copper, or lead gauze support, and baked. The negative pole consists of a plate of zinc, tin, or lead, and the whole is immersed in a solution of caustic soda contained in a vessel preferably made of iron.—B. T.

Dry Galvanic Cells. C. H. Mehner, Berlin, Germany. Eng. Pat. 7597, May 7, 1889. 4d.

A "MINERAL gelatin," which is in itself an excitant, is combined with the exciting solutions to form a pasty jelly-like mass suitable for a dry cell. This mineral is formed by adding magnesia to a concentrated solution of chloride of ammonia.—B. T.

Improvements in and connected with Electric Batteries. W. J. S. Barber-Starkey, Bridgnorth. Eng. Pat. 7619, May 7, 1889. 6d.

PLASTER of Paris is mixed with a suitable salt, such as the sulphates of sodium, potassium, or magnesium, soluble in the electrolyte, the mixture is quickly gauged and, while in the plastic state, placed in the cells between the plates. Upon the addition of water or the electrolyte the salt is dissolved out, leaving a very porous mass of plaster capable of absorbing a considerable quantity of the electrolyte and, at the same time, keeping the plates in one unalterable position. Porosity may also be gained by using some finely-divided fibrous material instead of the salt.—B. T.

XII.—FATS, OILS, AND SOAP MANUFACTURE.

Manganese Oxalate as a Drier. J. Castelaz. Chem. Ind. 12, 163.

MANGANESE oxalate, best obtained by treating the carbonate with oxalic acid solution, on heating with an oil to about 150° decomposes into carbonic acid, carbonic oxide, and oxide of manganese. By such treatment the oil gains in a high degree the capacity of drying. Whilst most of the driers hitherto used, when boiled with the oils, colour the latter, manganese oxalate can be used so as to yield almost colourless drying oils.—W. S.

Fatty Oil from Cyperus Esculentus. C. Hell and S. Twerdomehoff. Ber. 22, 1742—1745.

THE tubers of *Cyperus esculentus* contain, besides sugar, a considerable amount of fatty matter. The fat was extracted by means of petroleum ether, and was found to average 27.1 per cent. of the tubers. The oil is fluid at ordinary temperatures, yellow in colour, and possesses a smell somewhat similar to that of burnt sugar. When cooled below zero, a small quantity of solid glyceride separates. On saponification, the fatty acids were found to consist of oleic and myristic acids only, the bulk being oleic acid. The oil, therefore, seems to consist of the glyceride of oleic acid mixed with a little myristic glyceride.—H. T. P.

PATENTS.

Improvements in the Manufacture of Soap and Detergents. J. J. Bowley, Battersea. Eng. Pat. 9857, July 6, 1888. 4d.

THE object of this invention is to render soap more cleansing and non-corrosive to the material washed. For this purpose kerosene, petroleum, or shale oil is added to ordinary soap.—E. J. B.

Improvements in Soap and Plaisters. Edith Emily Gillam, South Kensington. Eng. Pat. 10,294, July 16, 1888. 4d.

THE object of this invention is to produce a disinfecting, antiseptic, anti-rheumatic, and antifebrile soap, by mixing together ordinary curd soap and from 10 to 20 per cent. of eucyput oil.—E. J. B.

Cooling or Refrigerating Lubricating Oils, and Apparatus therefor. D. Crawshaw, Sale. Eng. Pat. 10,803, July 26, 1888. 6d.

WHEN heavy bearings are lubricated, the practice is to cause a copious flow of oil through the journal, the escaping oil being collected and pumped back to the journals. The re-use of the oil is open to the objection that it soon becomes excessively heated, and thus loses its viscosity and lubricating power. This is obviated by the use of a cooler consisting of a number of tubes immersed in cold water.

—E. J. B.

Improvements in or appertaining to Composition or Soap used for Cleansing and Disinfecting Purposes. J. Crossley, Liverpool. Eng. Pat. 12,001, August 20, 1888. 4d.

THE improved composition consists of a mixture of soda crystals or ash, ordinary soap, and petroleum.—E. J. B.

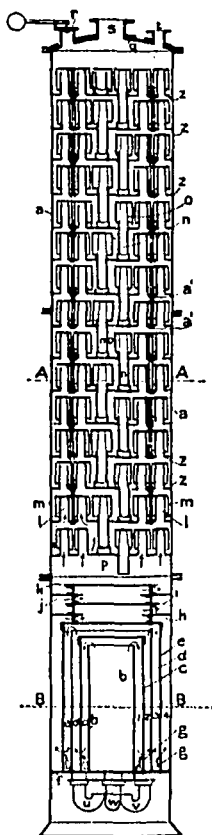
A Blue Soap for Laundry Washing. G. R. B. Kempton, West Ham. Eng. Pat. 5680, April 3, 1889. 4d.

THE object of this invention is to produce a blue soap, the use of which renders the subsequent employment of blue in laundry work unnecessary.

The inventor incorporates with ordinary soap a solution of aniline green in strong acetic acid. By the action of the alkali of the soap, the green is converted into blue, uniformly colouring the mass.—E. J. B.

Improved Apparatus for Rectifying Glycerin. R. O. Unglaub, Pendleton. Eng. Pat. 8196, May 16, 1889. 6d.

GLYCERIN vapour is made to pass through the cylinder A in the direction of the arrows. It is first made to



traverse the cylinders *b, c, d, e*, at the tops of which are the baffle plates *h, i, j, k*. Above these cylinders is a series of pipes *l*, fitted at the top with bonnets *m*. There is also a double row of pipes *n*, fitted with bonnets *o*, and traps *p*. The upper end of the cylinder is furnished with a safety valve *r*, opening inwards, and a pipe *s*, for drawing off the vapour.

The action of the apparatus is as follows: The impurities in the glycerin vapour condense on the surface of the cylinders, baffle plates, and pipes, and fall down to the bottom of the cylinder, where they are run off by the pipes *u, v, w*, the purified vapours being drawn off at the top and subsequently condensed.—E. J. B.

XIII.—PAINTS, PIGMENTS, VARNISHES, AND RESINS.

On the Green Ultramarine. J. Szilasi. Ann. **251**, 97—114.

ACCORDING to the researches of J. Philipp, R. Hoffmann, and K. Heumann, blue ultramarine is a definite chemical compound. The author has tried to prove first by analysis and afterwards by preparing derivatives from the green ultramarine that the latter also is a chemical compound and not a mixture. He analysed three kinds of this substance obtained from two different sources. Under the microscope they appeared entirely homogeneous, they contained no free sulphur or iron, and were free from other admixtures. The results of the analyses were remarkable for the fact that all three agreed very well together. The author describes further some derivatives prepared from the *Green ultramarine*, *Silver ultramarine*, *Lead ultramarine*, and *Zinc ultramarine*. From these experiments he draws the conclusion that green ultramarine is a definite chemical compound.—A. L.

Painting on Cement Plaster. Sels. Chem. Zeit. **13**, 696.

If paint be applied direct to plaster the caustic alkalis in the latter destroy the paint and make it peel off. Sulphate of iron (copperas) has been used to effect the so-called "killing" of the cement, the sulphuric acid of the copperas converting the caustic alkali or lime into sulphates, and thus destroying alkalinity, but a more efficient treatment is to soak the plaster with linoleic acid. When plaster so treated becomes dry any paint can be used with impunity.—E. E. B.

PATENTS.

New or Improved Composition or Liquid applicable for Preventing the Accumulation of Scale in Steam Boilers. J. W. C. Hamilton, Liverpool. Eng. Pat. 9324, June 26, 1888. 4d.

THE composition is made by adding from two to eight ounces of the following mixture to one gallon of purified paraffin.

	Per Cent
Essential oil of citronelle	40
" " cassia	25
" " lemon	9
" " eucalyptus	17

In some cases about four per cent. of crude camphor may be added.—E. J. B.

An Improved Method or Process of Treating Steel, Iron, or any Combination of such Metals, to Prevent their Oxidising, Rusting, or Corroding. T. W. Helliwell, Brighouse. Eng. Pat. 9570, July 2, 1888. 4d.

THE metal to be treated is heated "to about 600 degrees," whereby its "pores are opened," and immersed in a mixture composed of six pints of boiled oil, one "pint of fat," and one "pint of turpentine" for 5—15 minutes, then removed and either allowed to dry spontaneously or in an oven at "400 degrees." A second coating may be applied if desired. Metal treated thus is said to be protected from rusting or corrosion.—B. B.

Improved Coating or Preservative Material for Iron, Steel, and other Structures, or for Coating the Bottoms of Iron and Steel Ships or other Vessels, and Process for Manufacturing the same and other Products from Gas Tar. J. H. Eastman, Liverpool. Eng. Pat. 10,192, July 13, 1888. 4d.

Gas tar is freed from "ammonia and certain acids" alleged to militate against its employment as a preservative coating, by agitating it, heated to about 100° F., with water and calcium sulphate in the proportion of 20 gallons of water

and 10 lb. of calcium sulphate to 100 gallons of tar. The phenols form line compounds, and the ammonia yields ammonium sulphate; the former are recovered by acidulating with sulphuric acid and extracting with light tar oils, and purified by re-solution in caustic soda, removal of the neutral oils, and reprecipitation by sulphuric acid; the latter is obtained from its aqueous solution by evaporation.

The purified tar is mixed with one-tenth of its bulk of light petroleum, or coal-tar naphtha, and used as a preservative material for iron and steel.—B. B.

An Improved Compound for removing Paint from Painted Surfaces. P. Brentini, London. Eng. Pat. 11,398, August 7, 1888. 4d.

Four pounds of Irish moss, 3 lb. of methylated spirit, and 3 lb. of fuller's earth are mixed with 30 lb. of water, the whole boiled, and a solution of 16 lb. of caustic soda and 16 lb. of caustic potash dissolved in 28 lb. of water added, after which the product is stirred until it is cold and has solidified to a brownish gelatinous mass. The proportions of the ingredients may be varied. The compound is used by applying it to the painted surface with a brush, allowing it to remain thus for 20 minutes to one hour, and then washing it off together with the paint that has been disintegrated by its action.—B. B.

An Improved Varnish for Cleaning and Preserving Harness and other Leather Goods. S. B. Beswick, Birkenhead. Eng. Pat. 11,579, August 11, 1888. 4d.

Four ounces of shellac, half an ounce of camphor, and one ounce of rosin are dissolved in one pint of methylated spirit and shaken at intervals for 48 hours. The mixture is then coloured according to the kind of leather with which it is to be used. Other rosins, solvents, and proportions may be adopted.—B. B.

XVI.—SUGAR, STARCH, GUM, Etc.

The Amylodextrin of W. Nägeli, and its Relation to Soluble Starch. H. T. Brown and G. H. Morris. Chem. Soc. J. (Trans.) 55, 449—461.

AMYLODEXTRIN was first described by W. Nägeli in 1874. He obtained it by the long-continued action of dilute acids upon ungelatinised starch in the cold, when the residue was found to consist of amylodextrin. It was obtained in a pure state by treating this residuum with hot water and precipitating the solution with alcohol. Nägeli describes amylo-dextrin as separating from its solutions in crystalline spherules, made up of minute needles arranged radially. He assigned to the pure substance a rotatory power of $[\alpha]_{175} 175^\circ$ — 177° , and stated it to be non-diffusible. Of late years chemists have taken it for granted, mainly on the authority of Musculus and Gruber, and of A. Meyer, that amylo-dextrin is identical with soluble starch and the so-called starch cellulose.

The authors have now submitted this substance to a careful examination, and whilst failing to confirm in all respects Nägeli's statement regarding its properties, they have shown that it is a body of definite chemical composition, and have determined its relation to soluble starch, with which it certainly is not identical.

The authors show that when ungelatinised potato starch is treated in the cold with hydrochloric or sulphuric acid of about 10—12 per cent., little or no change can be observed in the microscopical appearance of the granules during a period of about 21 days. Notwithstanding this fact, the starch has been profoundly modified in its properties. Within about 24 hours of the commencement of the experiment the granules are found to have lost their power of forming a viscid paste with hot water; they dissolve to a perfectly limpid solution, and the substance which separates out on evaporation and cooling is found to have all the

properties of the soluble starch prepared by the limited action of diastase or dilute acid upon starch-paste at elevated temperatures. It is entirely without structure, and in the solid state has no influence on polarised light. It is coloured intense blue by iodine, both in solution and in the solid state, and it has a specific rotatory power in solution of $[\alpha]_{34.1} = 216.0^\circ$, and no cupric reducing power. When the digestion with acid has proceeded for a few days, the altered starch substance is found to have a gradually decreasing optical activity, whilst at the same time it acquires the property of reducing Fehling's solution, a property which goes on steadily increasing up to a certain point. Meanwhile the iodine reaction of the granules changes from a deep blue, through purple, reddish-purple, and reddish-brown to a final pale yellowish-red. After about three weeks' digestion the starch granules commence to disintegrate, and this action, which takes place principally along their lines of stratification, is complete in about three months. Chemical alteration of the residue continues for some time longer, but a point is ultimately reached beyond which no further change takes place.

The residue left after some months, representing about 60 per cent. of the starch originally taken, consists almost entirely of the so called amylo-dextrin. Purified by solution in water and precipitation with alcohol it gave the following numbers on analysis:—

$$[\alpha]_{34.0} = 206.25^\circ$$

$$K_{34.0} = 9.07^\circ$$

The authors then show that its composition can be expressed in terms of maltose and dextrin; it thus conforms to the rule previously established by them (Chem. Soc. J. (Trans.) 47, 538) for all starch transformation products. Its composition is:—

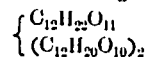
Maltose	-	14.87		
Dextrin	-	85.13	which requires	$[\alpha]_{34.0} = 205.4$
		100.00		$K_{34.0} = 9.07$

Brown and Morris state that one of the best marked properties of amylo-dextrin is that of forming spherocrystals, which are very beautiful in form when the substance is slowly separated out from solution. These spherocrystals resemble in their appearance so closely those of inulin that it is impossible by mere microscopic examination to distinguish between the two. That amylo-dextrin is really a homogeneous substance, and not a mixture, was proved in the following ways:—

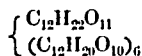
- (1.) It is absolutely unfermentable, and therefore cannot contain free maltose.
- (2.) It cannot be differentiated by fractionation of its solutions with alcohol, nor by methods of partial solution.
- (3.) The same is true with regard to dialysis. Contrary to Nägeli's statement, the authors find that amylo-dextrin is sensibly diffusible, and passes through the dialyser unchanged.

These facts, coupled with its distinctly crystalline nature, convinced the authors that they had to do with a perfectly definite substance, and a critical examination of the various stages in the production of this substance showed that it is derived from the partial or limited hydrolysis of soluble starch, the first product in the reaction. This partial hydrolysis of the starch ceases when a certain point is reached, the final product being amylo-dextrin.

Amylo-dextrin closely resembles in constitution and properties the malto-dextrin described by the authors in 1885 (Chem. Soc. J. (Trans.) 47, 538). Thus when treated with diastase or malt-extract both these substances are completely hydrolysed to maltose, whilst the authors have shown in their previous papers that starch, even under the most favourable conditions, leaves a residue of one-fifth of highly stable dextrin. Malto-dextrin was represented by Brown and Morris as consisting of one amylin- or maltose-group united to two amylin- or dextrin-groups. Thus:—



Amylodextrin can be represented in the same manner, and the authors' results show that it is made up of *one* amylo- or maltose-group united to *six* amylin- or dextrin-groups. Thus :—



Calculated.

Found.

$$[\alpha]_{583} = 206 \cdot 11^\circ$$

$$206 \cdot 25^\circ$$

$$K_{383} = 9 \cdot 08^\circ$$

$$9 \cdot 07^\circ$$

whilst a determination of the molecular weight by Raoult's method gave—

$$\Lambda \text{ (coefficient of depression)} = 0 \cdot 0086$$

$$M \text{ (molecular weight)} = 2220 \cdot 0$$

The above formula requires $M = 2286$.

Amylodextrin is differentiated from the two substances with which it has been confounded—soluble starch and the so-called starch cellulose—by its solubility, its iodine colouration, its crystalline structure, and its behaviour with diastase and with acids.—G. H. M.

The Determination of the Molecular Weights of the Carbohydrates. Part II. H. T. Brown and G. H. Morris. Chem. Soc. J. (Trans.) 55, 462—474.

THE authors first draw attention to the fact that the results given in their first paper on this subject have been confirmed by Tollens and Mayer (Ber. 21, 1566 and 3508), and by de Vries (Compt. Rend. 106, 751); they then give their later experiments in which a thermometer graduated to one-fiftieth of a degree, and capable of being accurately read to $0 \cdot 004^\circ C$, was used.

Galactose, $C_6H_{12}O_6$, $M = 180$.

—	Calculated for $C_6H_{12}O_6$	Found (Mean).
Λ (coefficient of depression) ..	$0 \cdot 100$	$0 \cdot 1073$
M (molecular weight)	$180 \cdot 0$	$177 \cdot 0$

Inulin, $n C_6H_{10}O_5$ or $C_{36}H_{62}O_{31}$.

It has been generally considered that the formula $n C_6H_{10}O_5$ expressed the constitution of inulin, but the recent researches of Kiliani indicate that a formula whose simplest expression is $C_{36}H_{62}O_{31}$ or $6 C_6H_{10}O_5 + H_2O$ is more probably correct. The numbers obtained by Raoult's method were :—

—	Calculated for $2 C_{36}H_{62}O_{31}$	Found (Mean).
Λ	$0 \cdot 0030$	$0 \cdot 0083$
M	$1980 \cdot 0$	$2150 \cdot 0$

From all the facts, and the great similarity between inulin and amylo-dextrin (see preceding abstract) the authors attribute the following formula to the two substances—



Inulin, $M = 1980$.

Amylo-dextrin, $M = 2286$.

The amylin- and amylo-groups in the two compounds possess very different optical properties, and behave differently with regard to cupric reduction. The ultimate products of hydrolysis with dilute acids are also very different, being in the one case levulose, and in the other dextrose.

Amylo-dextrin, $\left\{ \begin{array}{l} C_{12}H_{22}O_{11} \\ (C_{12}H_{20}O_{10})_6 \end{array} \right.$, $M = 2286$.

See preceding abstract.

Malto-dextrin, $\left\{ \begin{array}{l} C_{12}H_{22}O_{11} \\ (C_{12}H_{20}O_{10})_2 \end{array} \right.$, $M = 990$.

This substance was described by the authors in 1885. Submitted to Raoult's method it gave the following numbers :—

—	Calculated for $\left\{ \begin{array}{l} C_{12}H_{22}O_{11} \\ (C_{12}H_{20}O_{10})_2 \end{array} \right.$	Found (Mean).
Λ	$0 \cdot 0191$	$0 \cdot 0197$
M	$990 \cdot 0$	$965 \cdot 0$

Starch, $n (C_{12}O_{20}O_{10})$.—The authors found it impossible to apply Raoult's method to starch-paste; solutions of soluble starch produced so slight a depression that no reliable results could be obtained; a number of concordant results pointed, however, to a molecular weight of 20,000 to 30,000. In order to ascertain whether the failure in this case was due to a high molecular weight, or to the inapplicability of the method to colloid substances, the authors examined an arabinic acid, having a rotatory power of $[\alpha]_D = + 61 \cdot 16^\circ$; this gives results pointing to a value for M of 717, thus rendering it most probable that the small influence exerted by soluble starch on the freezing point was due to its high molecular weight.

Indirect evidence of the size of the soluble starch molecule was then sought from an examination of the dextrins. In former papers the authors have shown that when starch is broken down by diastase, a resting-stage in the reaction is reached when the amount of dextrin present in the conversion corresponds to one-fifth of the weight of the starch taken, and they have also shown from this and other facts that the molecule of soluble starch must be five times the size of the molecule of this stable dextrin. Determinations of several preparations of this dextrin gave the following numbers :—

—	Calculated for $20 C_{12}H_{20}O_{10}$	Found (Mean).
Λ	$0 \cdot 0020$	$0 \cdot 0030$
M	$6480 \cdot 0$	$6221 \cdot 0$

Consequently the value of soluble starch would be five times this, namely, its formula would be $5 (C_{12}H_{20}O_{10})_{20}$, and its molecule weight 32,400.

The size of the starch molecule having been as far as possible determined in this indirect way, the authors turned their attention to the dextrins. It is usually considered that these substances constitute a polymeric series, and it was endeavoured to apply Raoult's method to the solution of this question. A number of the higher dextrins were prepared from starch transformations stopped at an early stage of hydrolysis, and examined. All the results obtained show that the freezing method affords no evidence of there being any difference in molecular size between the so-called high and low dextrins, the numbers being, in fact, almost identical with those given above.

From a consideration of the results obtained with soluble starch, and with dextrins of varying position in the series, the authors conclude that the evidence points to the conclusion that the dextrins are metameric, and not polymeric compounds, as was suggested by O'Sullivan in 1879. They therefore abandon their former working hypothesis of the hydrolysis of starch by diastase, and now consider the starch molecule to consist of four complex amylin-groups, arranged round a fifth similar group, which constitutes a molecular nucleus. When hydrolysis takes place this complex is broken up, four amylin-groups being liberated; these are capable in turn of undergoing further hydrolysis into one or more malto-dextrins, and ultimately into maltose, whilst the fifth amylin-group, which constituted the nucleus of the original molecule, resists the action of hydrolysing agents, and forms the stable dextrin of the No. 8 equation.

The authors therefore assign to each amylin-group of the five, the formula $(C_{12}H_{20}O_{10})_{20}$ corresponding to a molecular weight of 6,480; the molecule of soluble starch being thus represented by the formula $5(C_{12}H_{20}O_{10})_{20}$ which corresponds to a molecular weight of 32,400.—G. H. M.

Pentacetyldextrose. E. Erwig and W. Koenigs. Ber. **22**, 1464—1467.

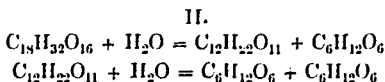
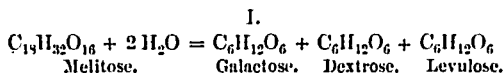
Up to the present time the only crystalline derivatives of grape-sugar known, which prove the presence of five OH-groups in the carbohydrate are aceto-chlorhydrate, $C_6H_5OCl(OC_2H_5O)_1$ and acetoneitrose $(OC_2H_5O)_4C_6H_7O(ON_2)$, neither of which crystallises very readily.

The authors have succeeded in obtaining a pentacetyldextrose by boiling pure grape-sugar (5 grms.) for 10 minutes with acetic anhydride (20—22 cc.), and a trace of zinc chloride; the yield is good.

As pentacetyldextrose crystallises well, and can be easily obtained in a pure state, its formation can be employed for the identification of grape-sugar.—F. S. K.

The Inversion Products of Melitose (Raffinose). C. Scheibler and H. Mittelmeier. Ber. **22**, 1678—1686.

According to the observations of different chemists, it seems probable that melitose yields on inversion a mixture of three glucoses: galactose, dextrose, and levulose. The inversion may take place either in one or two stages, as shown by the following equations:—



By the action of very dilute sulphuric acid, melitose is split up according to the first equation under II. That the reaction takes this course was shown by the fact that the osazone prepared from the inversion products could be separated into two portions, one soluble, the other insoluble in hot water. Now, according to E. Fischer, the osazones of the simple glucoses are all insoluble in hot water, whilst the diglucoses, such as milk sugar, yield an osazone soluble in hot water. The glucose formed was levulose. The diglucose produced has apparently the composition of milk sugar, but a closer examination proved it to be a new body. Its specific rotary power, as calculated from the optical activity of the products of inversion, is considerably higher than the specific rotary power of lactose. Its osazone is a yellow powder, melting, when quickly heated, at 176° — 178° C. Lactosazone melts at 200° C. The new osazone is fairly soluble in hot alcohol, but separates slowly on cooling. It is difficultly soluble in ether, chloroform, and benzene, easily soluble in warm strong acetic acid, forming a dark red solution. So far the authors have been unable to obtain the new sugar in the crystalline state. They propose to name it "melibiose." The complete inversion of melitose requires protracted heating with sulphuric acid, the solution becoming very yellow in colour, and a small quantity of humin substances being separated. The mixed osazones prepared from the products of inversion were practically insoluble in hot water, and melted at 200° — 201° C. (Compare this Journal, 1887, 445).—H. T. P.

The Oxidation of Rhamnose (Isodulcitol) by Nitric Acid. W. Will and C. Peters. Ber. **22**, 1697—1704.

MALIN obtained, by the oxidation of rhamnose with strong nitric acid, an acid of the formula $C_6H_{10}O_9$. This result is incompatible with the present view of the constitution of rhamnose, which is considered to be an aldehyde of the formula $C_6H_{12}O_5 + H_2O$. The authors have, therefore, re-investigated the subject, and find that the chief product

of the oxidation of rhamnose by nitric acid is trihydroxyglutaric acid. The potassium salt of the acid was prepared, and found to be in every respect identical with the potassium salt of trihydroxyglutaric acid, as described by Haushofer. The calcium salt, $C_5H_6O_7Ca + 3H_2O$, is prepared by mixing a strong solution of the potassium salt with calcium chloride. The barium compound is prepared in a similar way. On adding lead nitrate to a dilute solution of the potassium salt, a white amorphous precipitate of the lead salt is obtained. The silver compound obtained in a similar manner is a voluminous, colourless precipitate, rapidly changing to the crystalline state. The formation of this acid settles any doubts as to the constitution of rhamnose, and proves it to be $CH_2.CHOH.CHOH.CHOH.CHOH.CO.H$.

In a former paper the authors described a lactone, $C_6H_{10}O_5$, obtained by the action of bromine and water on rhamnose. This body was very similar to Kiliani's meta-saccharin. The authors have prepared crystals of the body, and find it to be totally different to meta-saccharin.

—H. T. P.

PATENTS.

Process and Apparatus for obtaining Sugar Liquor from Sugar Cane, or for obtaining Extracts. H. A. Hughes, Rio Grande, N.J., U.S.A. Eng. Pat. 7131, April 29, 1889. 8d.

THE main feature in this invention is the manner in which sugar cane, or sorghum, is subjected to the process of diffusion. In this case the diffusion battery consists of a series of shallow round cells placed in an annular tank provided with a cover, to which the cells are attached, the annular tank serving as a steam or hot-water jacket to the cells. By means of a crane with horizontal arms arranged round a vertical shaft placed in the centre of the battery system, baskets made of perforated sheet copper, and containing the cane chips, are placed in the cells, which contain water, in rotation, the chips being exhausted when they leave the last cell. When the liquor in the first cell is sufficiently dense it is drawn off and the cell refilled with water, the process being carried out in the well known systematic manner. To avoid the gradual removal of water from the first cell, if the chips were introduced dry, the baskets containing the chips are first dipped into another tank of strong sugar liquor before being introduced into the regular battery cells. Apparatus is also described for removing the leaves from the canes and cutting and shredding the stalks. Detailed drawings accompany the specification.—W. M.

Apparatus for Refining Loaf Sugar in the Moulds. C. Steffen, Vienna, Austria. Eng. Pat. 8052, May 14, 1889. 8d.

THE apparatus consists of "a series of moulds with or without sieves at their apex according to the kind of sugar used, that is sugar in hard crystallised form, or in the form of granular pulpy mass, arranged one above the other so as to form an air-tight fitting column-like battery, into the upper mould of which the lixiviating fluid, consisting of a saturated solution of pure sugar, is led, from whence the said fluid descends through all the moulds in the said battery, and leaves the last mould as an impure syrupy fluid, the moulds in the said battery being exchanged by removing the uppermost mould containing perfectly refined sugar, and inserting a fresh mould, charged with sugar to be treated, at the lowermost end of the battery."

Compressed air is used to force the liquor out of the uppermost mould when it has been sufficiently refined. Drawings accompany the specification clearly showing the mechanical carrying out of this process.—W. M.

XVII.—BREWING, WINES, SPIRITS, Etc.

PATENTS.

Improvements in the Rectification of Alcohol. A. T. Christophe, Paris, France. Eng. Pat. 12,642, September 1, 1888. 6d.

PREVIOUS to distillation the spirit is treated in the cold with sodium amalgam, or with an alloy of sodium with tin, lead, or other suitable metal. Calcium hypochlorite (bleaching powder) is also added. The operation is best conducted as follows: The spirit is placed in a large vat provided with stirring apparatus, and diluted with water to about 40 per cent. strength. Bleaching powder is then stirred in at the rate of 1 gram. per litre of the diluted spirit. After standing some minutes the sodium amalgam or alloy is added in such quantity that about 15–20 grms. of sodium are present per 100 litres of the spirit. The stirrers should be kept in motion for half an hour or more. After some time the spirit is transferred to the rectifier and distilled in the usual manner. It will be found that the good spirit comes over at an earlier stage than usual, and the yield is larger and better in quality.—H. T. P.

A Process for Producing Extract of Hops, and for the Preparation of a Beverage or Beverages therewith. L. Groezinger, Stuttgart, Germany. Eng. Pat. 13,212, September 12, 1888. 6d.

THE resinous matters are first removed by means of spirits of wine. The extraction is carried on in wooden or tinned vessels provided with false bottoms and taps for drawing off the liquor. The hops are tightly packed in and covered with four parts of alcohol to one of hops. After two days the extract is run off and the hops pressed. The hops are next boiled with water in a copper boiler. The hops are placed on three trays made of woven tin wire stretched over tin hoops. These trays rest on projections inside the boiler and can be removed at will. The pressed hops are shaken out and placed between the divisions of the boiler and boiled with 15 parts of water for every one part of original hops. After half an hour the extract is run off, and the extraction continued with a fresh portion of water, and so on. The decoction so made can be clarified with gelatin, albumen, &c., in the usual way. To make beer from this extract, malt-sugar and colouring matter are added, and the mixture is filtered and aerated with carbonic acid gas. The extract may also be employed to improve fermented beer at any stage of its preparation. For this purpose a stronger extract may be made by mixing the spirit infusion with the first and second decoctions, and taking only 6–8 parts of water in the preparation of these decoctions.—H. T. P.

Improvements relating to Apparatus for the Pasteurisation of Beer, Wine and other Liquids. W. Kuhn, Clermont-Ferrand, France. Eng. Pat. 15,261, October 23, 1888. 11d.

THE apparatus consists of a large copper cylinder surrounded by a sheet iron jacket or outer cylinder, the whole being arranged horizontally. The ends of the two cylinders can be easily taken off, so as to allow of the apparatus being cleaned. A large copper coil occupies the whole length of the inner cylinder. One end of the coil passes right outside the apparatus; the other end communicates with the space between the two cylinders. The apparatus is provided with a pressure gauge, two thermometers, and a number of taps for the entrance and exit of beer, gas, and heating and cooling water. The whole is supported on iron feet. The beer to be pasteurised is filtered and forced into the apparatus at the bottom. When full, the tap is closed, and hot water is circulated through the coil and jacket. When the beer has been brought to the desired temperature and pressure, it is again rapidly cooled by passing cold

incoagulable liquid through the coil. Pure air or carbonic acid gas is then forced into the apparatus, and the beer transferred to sterilised and hermetically closed casks. These cylinders may also be combined in sets of three, the liquid to be sterilised being heated in the first, partially cooled in the second, and completely cooled in the third. An arrangement is described in which the coils are placed vertically. It consists of three copper vessels superimposed and provided with coils, casings, &c. as before. The beer is heated in the top vessel, and passes through the middle and bottom vessels, in which it is cooled. A third apparatus is described, by means of which a continuous flow of sterilised beer may be obtained, without loss of gas. It consists of three vertical cylinders with coils, &c., each preceded by an additional coil. Between each set of two coils, and also at the beginning and end of the apparatus, there is a pump. Beer is forced continuously through the apparatus. It is sterilised in the first two vessels, and cooled in its passage over the second and third set of coils. No escape of carbonic acid can take place at any stage, for the pump-valves are so arranged that when one is open the preceding and following ones are closed.—H. T. P.

Apparatus for Supplying Carbonic Acid Gas to Liquors in Casks or other Receptacles. H. Hürter, Coblenz, Germany. Eng. Pat. 5940, April 6, 1889. 6d.

THE apparatus consists of a wrought-iron gas cylinder, open at one end, in which a valve is screwed. A narrow passage connects the reservoir with the valve chamber. A screw spindle passes through a stuffing box into the valve chamber. The spindle terminates outside in a triangular head, so that by means of a key it can be screwed right down on the narrow opening leading from the reservoir, closing it. The valve chamber is provided with a lateral opening, by means of which the reservoir is filled and emptied. To provide means for fixing the apparatus in cask, the nut at the top of the valve chamber carries a tube, which is screw-threaded outside. Having filled the cylinder through the side passage, the valve is screwed down and protective caps screwed on the lateral opening and over the valve. The apparatus may be kept filled for an indefinite time. To insert the apparatus in a barrel, the latter is provided with a screw bung, having an opening closed by a screw plug. The bung is unscrewed, the apparatus screwed in place of its inner plug, so that the head of the valve may be reached through the opening in the bung; the apparatus is passed into the cask, and the outer bung now carrying the apparatus screwed fast. By turning the spindle from the outside by means of the key, the gas will escape inside the cask.—H. T. P.

XVIII.—CHEMISTRY OF FOODS, SANITARY CHEMISTRY, AND DISINFECTANTS.

(A)—CHEMISTRY OF FOOD.

PATENT.

An Improved Manufacture of Cattle and other Food. A. W. Macellwaine, Kingston-on-Hull. Eng. Pat. 10,488, July 19, 1888. 4d.

By extracting the residue from the preparation of mustard from mustard seed, by means of volatile solvents and steam, the inventor renders it fit for admixture with cattle and other food.—E. J. B.

(B)—SANITARY CHEMISTRY.

PATENT.

Improvements in the Method of applying Soluble Disinfectants and Antiseptics, and in means therefor. R. Le Neve Foster, Manchester. Eng. Pat. 7602, May 7, 1889. 4d.

SOLUBLE disinfectants in a solid form, *e.g.*, carbolic, cresylic or other tar acids, are enclosed in a cage of wire work or perforated metallic sheeting, such cage being either floated on the liquid to be impregnated or fixed at the normal fluid level thereof.—C. C. H.

(C)—DISINFECTANTS.

PATENT.

Improvements relating to the Utilisation of Peat for Antiseptic and other Purposes. C. Kuttén, Griveprée, Belgium. Eng. Pat. 11,465, August 8, 1888. 6d.

PEAT has long been used as a dressing for wounds, but though it possesses surprising healing qualities it has not been largely used on account of its inconvenient character. The present invention remedies this by producing a peat wadding. After dessication the raw peat is heckled to remove knots, then beaten and mixed with a small quantity of cotton, hair, or wool, or other suitable material, to give more cohesion to the finished mass; it is carded. Finally, it is slowly boiled in water, with or without the addition of antiseptics, such as corrosive sublimate, and carded into a uniform nap of wadding; this renders it soft, pliable, and absorbent, without detracting from its valuable properties as a surgical bandage.—C. C. H.

(clay), liberating the silica, and combining with the alumina to form sulphate of alumina. Any lime, and indeed the greater part of the silicate of iron, also oxide of iron, which are present in the calcined clay, are also acted upon, forming soluble salts, which, as a matter of fact, constitute the impurities of aluminous cake. After the violent action has subsided, and the thick putty-like mass begins to get stiffer, the charge of clay and acid is dropped through a door in the bottom of the mixer into a large iron waggon with moveable sides, where it is kept warm and allowed to remain for twelve or more hours to enable the chemical action to cease. After the lapse of this time the moveable sides of the iron waggon are removed, and the mass of aluminous cake, which is still hot and comparatively soft, is loosened, and by means of a screw worked automatically with suitable machinery, is pushed forward about half an inch at a time underneath the knife of a huge guillotine, which cuts the face of the block in a vertical direction as the knife descends, the aluminous cake falling in fragments into a waggon placed immediately underneath. The cut cake is then stored in heaps until it appears dry, or until all further possibility of the acid acting upon the clay has ended. In this state it is placed in bags and sold to the paper makers. Its composition is fairly regular, and may be represented by the following analyses of the English-made article:—

ANALYSES OF ALUMINOUS CAKE.

—	"A."	"B."
Sulphate of alumina	Per Cent. 41·80	Per Cent. 39·40
Free sulphuric acid.....	·81	2·65
Sulphate of peroxide of iron	·45	·50
Silica, undecomposed clay, &c., or substances insoluble in water	24·90	20·48
Water, lime, magnesha, loss, &c.	32·01	36·97
	100·00	100 00

XIX.—PAPER, PASTEBOARD, Etc.

Aluminous Cake: Its Composition and Use in Paper Making. Chemical Trade Journal, 5, 158.

SULPHATE of alumina is extensively used in the sizing of paper pulp in the beater engine. There is a large home consumption of aluminous cake, and also a large export trade between this country, the Continent, and America. Its application is confined to the manufacture of cheap printing papers. For finer grades of paper it might also be used providing not too much per ton of paper were required and that it were free from iron. Its mode of preparation is as follows:—

The china clay is first of all calcined by being heated on the bed of a reverberatory furnace until all hygroscopic, and practically the whole of the combined water has been driven off. The calcined clay is now withdrawn and cooled, and consists essentially of an anhydrous silicate of alumina, together with other substances which, in our particular case, may be looked upon as impurities, namely, a small percentage of lime, and a little silicate of iron, a portion of which, however, is doubtless decomposed during the calcining operation. The calcined clay is then removed when cold, and is finely ground in a mill suitable for the purpose, the ground portion being riddled upon a very fine sieve. The coarse granules of clay, which do not pass through the sieve, return to the mill and are re-ground, whilst the sieved portion is used for mixing with the oil of vitriol. This mixing of the clay with the oil of vitriol is the most important operation in the preparation of aluminous cake, and is generally carried out according to exact measurement in large cast-iron vessels or vessels of other material lined with lead and fitted with agitators with which the mixture is stirred. The clay is introduced into the "mixer" simultaneously with the acid in suitable proportions, the acid being of suitable strength. A very violent reaction takes place, in which the sulphuric acid splits up the silicate of alumina

The quality of "A" is superior to that of "B," because it contains considerably more sulphate of alumina, less free sulphuric acid and sulphate of peroxide of iron, and more insoluble matter. The amount of free acid in "B" is very high and is really unusual. Where it contains this ingredient in such quantity the aluminous cake is somewhat soft and appears damp and dull in colour.

Aluminous cakes seldom contain much sulphate of lime, and if they do, this impurity does not affect their applicability in the manufacture of paper. Nor, indeed, does the presence of a small quantity of free acid such as that contained in "A," because it can always be neutralised by the soda contained in the resin size used along with aluminous cake in the process of sizing paper pulp. Excepting dirt, the sulphate of peroxide of iron (ferrie sulphate) is the only impurity which the cake usually contains, and it is this towards which the paper maker has such an objection. As a matter of fact, however, the purposes for which aluminous cake is used are such as not in most cases to be influenced by the presence of a small quantity of sulphate of iron like that which exists in ordinary aluminous cake. The iron salt will, of course, have a telling effect upon the finished paper, should it be present in abnormal quantity, say to the extent of four or five per cent., but as a general rule it does not contain anything like this quantity. The amount of anhydrous sulphate of alumina which it usually contains is from 40 to 42 per cent. (equal to from 11·93 to 12·52 per cent. of alumina (Al₂O₃)). This sulphate is the real sizing material, and that for which the cake is used, and in practice its percentage amount should always be regular or within certain limits.

The distinguishing feature of aluminous cake when compared with other aluminous compounds used by paper makers for sizing, lies in the fact that it always contains

a certain percentage of silica and undecomposed clay which are insoluble in water, but serve as loading material when it is added in the crude solid state to the pulp in the beater engine. In the manufacture of common news- and cheap printing-papers, when loading substances are used, this insoluble matter in the aluminous cake remains with the fibre, and appears in the finished sheet of paper, representing so much ash.

By virtue of this fact, the insoluble matter possesses a value of its own, corresponding to its equivalent in weight and the value of china clay, which, as is well known, is the most commonly used loading material in the paper trade.

Aluminous cakes should be free from grit and dirt of any kind, and it is usually added directly to the pulp in the beater engine, no care on the part of the paper maker being taken to free it from these impurities before use. The insoluble matter consists of varying quantities of silica and undecomposed clay. Although these are in a fine state of division, they do not possess the same properties as china clay. Neither of them feel so soft or soapy when rubbed between the fingers. The silica is in a state of very fine powder, but nevertheless does not possess the power of communicating that body and surface frequently required in finished paper, and any lack of surface on papers sized with aluminous cake is very likely due to the presence of this ingredient. The requirements of the printer demand that paper, when used on the high-speed printing machines of the present day, will not yield any dust, and as it is difficult to fix silica within the fibre when in a very fine state of division on soft size paper, especially when starch cannot be or is not used, the probability is that this silica, and indeed the undecomposed clay as well, is thrown off on unwinding the reel as it enters the printing machine and causes the trouble referred to, besides stopping up the type. A common basis of comparison of the amounts of ferric sulphate in aluminous cakes offered in the market, is generally adopted. If the amount of sulphate of iron be calculated on 100 parts of anhydrous sulphate of alumina in each of the analyses of the foregoing samples, the relative quantities of iron salt are strictly comparable. Anhydrous sulphate of alumina is really the useful ingredient to paper makers in the aluminous cakes they buy, and hence to compare one sample with another, all impurities should be calculated on, say, 100 parts of sulphate of alumina. In the case of the impurity, viz., ferric sulphate, in analysis "A" we have thus, 1.076 parts, and in "B" 1.269 parts on 100 of anhydrous sulphate of alumina. This example may be extended of course to comparison with the other impurities.—W. S.

PATENTS.

Bleaching Paper Pulp by Electricity. E. H. M. Andreoli, Brixton. Eng. Pat. 8161, June 4, 1888. 6d.

THIS process relates to the bleaching of paper pulp, &c. by means of an electrolysed solution of common salt or sea water. The inventor claims in addition: The construction of anodes and cathodes by fusing, casting, reducing, and peroxidising chloride of lead alone (for cathodes) and in combination with other metallic salts, such as chloride of manganese (for anodes); the use of an alternating current: the treatment of paper pulp in bleaching tanks where the electrolyte, raised to its proper degree of bleaching power by the action of oxygen or ozone in its nascent state, in combination with chlorine and hydrogen, circulates and is maintained at the required chlorometric standard by the influx of freshly electrolysed solution to replace the corresponding quantity of exhausted electrolyte which flows out of the tank by a pipe, and also of a large peroxidised anode and a spongy lead or carbon cathode through which an electric current passes. He also claims the whole process for bleaching paper pulp by the action of hypochlorite of sodium electrolytically generated by means of plates made of a cheap conductive material not easily attacked and corroded by chlorine or oxygen, such as wood charcoal, either for both cathode and anode, but specifically for anode, in the electrolytic tank where chlorine is generated

and combines with hydrogen and oxygen in a more or less active state. It is advisable to heat the liquid to be electrolysed to 60° C.—E. J. B.

Improvements in Machinery or Apparatus for Coating Paper or other Fabric with Glue, Gum, Paste, or other Fluid or Semi-fluid Material. G. J. Feldon, Lambeth. Eng. Pat. 18,066, December 11, 1888. 8d.

A CYLINDER is fixed upon a shaft mounted so as to revolve in a bath of the coating material, so that the underside of the periphery of the cylinder is immersed in the bath. The fibre to be coated is drawn across the cylinder from between nipping rollers covered with india-rubber.—E. J. B.

XX.—FINE CHEMICALS, ALKALOIDS, ESSENCES AND EXTRACTS.

Myrtle Oil and Myrtol. E. Jahns. Arch. der Pharm. 27 [3], 174.

THIS oil as used medicinally begins to boil at 160° and about 80 per cent. distilled over up to 240° C. The residue consisted of high-boiling terpenes partly resinised and partly polymerised. From the portion boiling between 160° to 240° C., by repeated fractional distillation the following constituents were separated:—

A Terpene, $C_{10}H_{16}$, boiling between 158° and 160°, and having a specific rotation $[\alpha]_D = +36.8^\circ$. Its properties led the author to believe it to be right-handed *Pinene*.

Cineol, $C_{10}H_{18}O$, which purified by Wallach's method, boiled constantly at 176°.

A *Camphor*, probably of the formula $C_{10}H_{16}O$, in small quantity, boiling at 195° to 200° C. The therapeutic value of the *Myrtle Oil* is to be traced to the amount of cineol present in it, the latter substance being identical with eucalyptol and eucalyptol.—W. S.

Some New Compounds of the Quinine Alkaloids. J. Hesse. Pharm. Zeit. 1889, 191.

THE compound of phenol with some salts of the quinine alkaloids of left-handed polarisation have long been known and are much used as febrifuges, as e.g., quinine phenol sulphate, $(C_{20}H_{21}N_2O_2)_2SO_3 \cdot C_6H_6O + 2H_2O$. The author has recently prepared a new compound of phenol with quinine bisulphate. If carbolic acid be added in equivalent proportion to a hot aqueous solution of quinine bisulphate, on cooling at first an oily mass separates out, above which delicate white needles are gradually formed, of the composition $C_{20}H_{21}N_2O_2 \cdot SO_3 \cdot C_6H_6O + 3H_2O$. This compound is very unstable. If it be dissolved in hot water, on cooling the first-named normal salt crystallises out. Quite in accordance with this is the behaviour of the acid Quinine resoreinol sulphate, the acid Quinine quinol sulphate, the acid Quinine pyrogallol sulphate, and the acid Cinchonidine pyrogallol sulphate, for one and all they separate out the normal salts when the attempt is made to recrystallise them from hot water. The author further prepared and describes the normal Quinine oreinol sulphate, Quinine catechol sulphate, Cinchonidine quinol sulphate, as well as the Quinine resoreinol, Quinine catechol, and Quinine pyrogallol hydrochlorides. They are beautifully crystallised compounds and some of them excellent febrifuges.—W. S.

Mercury Benzoate. E. Lieventhal. Pharm. Zeit. Russl. 28, 310.

One hundred and twenty-five parts of mercuric oxide are dissolved in 250 parts of nitric acid (1·20 sp. gr.), the solution diluted with 4,000 parts of water and filtered; to this a solution of 188 parts of sodium benzoate in 4,000 parts of water is gradually added with agitation. The voluminous precipitate of mercuric benzoate obtained, after washing, pressing and drying, forms a light, white powder, soluble with difficulty in ether, alcohol, chloroform and water.—O. H.

Macassar Oil. L. v. Italic. Apoth. Zeit. 1889, 4, 506.

Schleichera trifuga is one of the *Sapindaceæ* and bears red-brown seeds averaging 0·5 grm. in weight; these contain about 36 per cent. of a buttery fat which is "Macassar oil." It has a specific gravity of ·924 at 15° C., melts at 28° C., has an iodine number of 53, a saponification equivalent of 219 (1 grm. requires 230 mgrm. of potash for saponification), contains 91 per cent. of insoluble fatty acids and 6·3 per cent. of glycerol. The fatty acids present include acetic, butyric, lauric, arachidic and oleic acids.

—B. B.

Macassar Oil. K. Thümmel. Apoth. Zeit. 1889, 4, 518.

A SAMPLE examined by the author had a melting point of 21°–22° C. instead of 28° C. given by *Italic* (see preceding abstract). The presence of hydrocyanic acid was detected and 0·047 per cent. obtained by steam distillation. Benzaldehyde was recognised in the distillate by its transformation into benzoic acid by the action of potassium permanganate. These substances might have owed their origin to sophistication with bitter almond oil; an examination of the seeds would settle this point.—B. B.

Compounds of Mercury with Phenols. E. Merck. Apoth. Zeit. 1889, 4, 651.

Mercury phenate, Hydrargyrum phenolicum, is obtained by precipitating corrosive sublimate with sodium phenate. According to the conditions of precipitation a more or less basic salt is obtained which is used for injections in the treatment of syphilis.

Mercury thymylate is obtained by precipitating mercuric nitrate with sodium thymylate and forms a greenish-violet precipitate of the formula $C_{10}H_{13}HgOH$.—E. E. B.

Distilled Perfumes and Essential Oils. Report by Consul Mason, United States Consulate, Marseilles, 602.

THE process of distillation varies but slightly for all varieties of lavender, and the same apparatus is often used successively for each kind of plant as its season of flowering and harvest arrives. The necessary machinery includes as its principal feature a copper alembic, usually about 6 ft. in height by 4 in. diameter, the neck of which is carried over and terminates in a spiral coil immersed in cold water. Each alembic has, near the top, an opening closed by a man-head, secured by bolts as in ordinary steam machinery. Through this opening the mass of fresh plants is packed into the interior until it is completely filled. Near the bottom of the alembic another similar opening is provided through which the spent material may be withdrawn with a pronged hook like a manure fork with curved tines. The bottom is covered with a perforated copper diaphragm, under which lies a flat coil of pipe, likewise perforated, through which steam is introduced from a boiler in which a pressure of from 5 to 7 atmospheres is maintained. The alembic being thus charged with raw material—either freshly gathered or dry—and the man-heads closed, the steam is turned on and forces its way upward through the mass, absorbing and carrying the perfume over into the submerged coil, where the oil condenses and trickles out with the distilled water, upon which it floats by reason of

lighter gravity. In about three hours the perfume is exhausted, when the alembic is emptied and recharged. Three hundred pounds of dried lavender plants, or 220 lb. of *Aspic*, are required to produce 1 lb. of essential oil.

There are two species of the absinthe plant—the large and small—which are used respectively in the manufacture of absinthe liqueurs and vermouth. In manufacturing the liqueur the upper leaves and twigs of the plant are macerated with hyssop, calamus, citronelle, anise, fennel, diadane, and other vegetable substances. The decoction thus obtained is distilled, and the product treated with alcohol, sugar, and various colouring matters.—A. J. S.

Report on New Drugs and Fine Chemicals. Merck's Bull. 2 [6].

Chromic Acid.—This acid has been known as an escharotic in the nasal and pharyngeal cavities as far back as 1885. Danger had hitherto been found in the use of this stringent means of topical causticisation, of cauterising the adjacent healthy parts. This danger has been traced to the usual presence of sulphuric acid in the preparations offered. This sulphuric acid makes the chromic acid (anhydride) deliquescent and causes it therefore to run upon adjacent parts and so spread the action too far. If but traces of sulphuric acid are present the action may be injurious, and hence the Prussian War Department ordains that only chemically pure chromic acid may be used in the treatment of foot-sweat among the troops.

Pyrodine or Acetyl-phenyl-hydrazine.—Guttmann's researches (Berliner Med. Ges. May 1, 1889) fully confirm the results of Dreschfeld of Manchester.

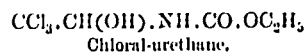
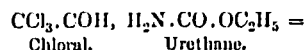
Antiseptine (Brominated Antifebrine or Parabromo-phenyl Acetamide), $C_6H_4(Br)NH.COCH_3$, is devoid of smell or taste. It crystallises in small, pearly prisms, melting at 164·5° C. It is insoluble in cold, with difficulty in hot water, only slightly in glycerol, but easily in alcohol or ether. A one per cent. alcoholic solution may be treated with twice its volume of water or thrice its volume of glycerol, without turbidity ensuing.

Eralgine (Methylphenylacetamide or Methylated Antifebrine), $C_6H_5N(CH_3)CO.CH_3$.—It appears in the form of white plates or fine white needles, melting at 101° C. Compare this Journal, 1889, 412 and 567.

Methacetine (Oxymethylated Antifebrine or Para-acetanisidine), $C_6H_4(OCH_3)NH.COCH_3$.

As might have been suspected, from its chemical constitution, so analogous to that of Phenacetine, it appears that Methacetine has developed physiological properties quite similar to those of the former substance. Its action is antithermic, without untoward necessary symptoms, though frequently, according to Mahner, heavy perspirations ensued. The antiseptic properties of Methacetine are proved by the fact that a 1 per cent. solution arrests the decomposition of milk, and prevents the ammoniacal fermentation of urine.

Chloral Urethane, $CCl_3.CH(OH)NH.CO.OC_2H_5$ is obtained by the union of 1 mol. of chloral (not the hydrate) to 1 mol. of urethane, thus:—



It is a crystalline mass, insoluble in cold water, and decomposed by boiling water into its two components. Alcohol and ether dissolve it readily, and water separates it from these solutions. Its melting point is about 103° C.; it attains this point only, however, after partially decomposing from 100° C. upwards. Chloral-urethane was first investigated as to its therapeutic properties by Hübner and Sticker (*Deutsch. med. Wochenschr.* 1886 [14], 236), and compared with ethylidene-urethane and methyl-urethane. The last two compounds were found to be entirely inert in doses of 1

to 4 grms., whilst chloral-urethane produced hypnotic effects like ethylic-urethane, although in a less certain and persistent degree. Mairat and Combemale (*Montpellier méd.* 1886, 149) concluded that the toxic effect outweighed the hypnotic in prominence and importance of symptoms. The sleep induced was always attended with paralysis of the hinder parts in the case of animals.

Nicotine Bitartrate (Acid tartrate).—The manufacturing method for preparing this salt was invented by Dr. Dreser, of Strasburg. It is a beautifully crystalline substance, and perfectly stable, whilst on the other hand all the remaining known simple salts of nicotine are very difficult to crystallise, and hence the strengths of their solutions become variable and unreliable. Also the solutions of the free alkaloid have the tendency to resinify, thicken, and darken, and to become therapeutically unavailable. The *Bitartrate* (Dreser) crystallises in fine white tufts of needles, and is very readily soluble in water. Its composition, on analysis, shows the proportions $C_{10}H_{11}N_2$ (nicotine) = 32.53 per cent.; 2 $(C_4H_4O_6)$ tartaric acid = 60.51 per cent., and 2 H_2O = 7.34 per cent. The physiological experiments (*Archiv. der Pharm.* 1889, 269) showed that the well-known specific effects of the pure alkaloid are all produced likewise by this salt, and in like degree.—W. S.

Synthesis of Active Coniine. A. Ladenburg. *Ber.* 22, 1403—1405.

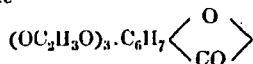
SCHÖLLEMMER ("Rise and Development of Organic Chemistry") has stated that the complete synthesis of active coniine has not yet been accomplished, but the author has previously shown that the alkaloid in question can be obtained from its elements.

Acetic acid can be prepared synthetically, and from it acetone, isopropyl alcohol, and glycerin can be successively obtained. Glycerin can be converted first into bromallyl, and then into trimethylene bromide, and from the latter piperidine and finally pyridine can be produced. α -Picoline can be obtained from pyridine (Ladenburg and Lange, *Ann.* 247, 5), and can be converted into coniine (Ladenburg, *Ann.* 247, 80). (*This Journal*, 1887, 224.)

The dextro-tartaric acid, which is necessary to the production of the active base, can also be prepared from its elements.—F. S. K.

Acetyl Derivatives of Quinic Acid. E. Erwig and W. Koenigs. *Ber.* 22, 1457—1464.

Triacetylquinide—



is obtained when quinic acid (10 grms.) is boiled with acetic anhydride (70 cc.). After distilling most of the acetic anhydride and acetic acid the residue is repeatedly evaporated with absolute alcohol on the water-bath, washed with cold ether to remove resinous impurities, and recrystallised several times from boiling alcohol with addition of animal charcoal. The yield of pure substance is 8 grms. Triacetylquinide melts at 132° , is almost insoluble in cold dilute sodium carbonate and only sparingly soluble in cold water and cold alcohol, but readily in the boiling solvents. The conversion of quinic acid into the crystalline and easily-purified triacetylquinide can be employed as a most characteristic reaction for the identification of small quantities of the acid.

When triacetylquinide or quinic acid is heated with acetic anhydride at 240° an isomeric triacetylquinide (isotriacetylquinide) melting at 139° is formed. The yield of pure substance, isolated as described above, is 50 per cent. of the quinic acid employed. Isotriacetylquinide is almost insoluble in cold dilute sodium carbonate, and is similar to triacetylquinide in its behaviour towards solvents.

When quinic acid is boiled with acetic anhydride and sodium acetate the principal product is the triacetyl derivative, melting at 132° , but when a small quantity of anhydrous zinc chloride is employed instead of sodium acetate, tetracetylquinic acid is formed in almost theoretical quantities.

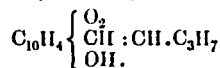
Tetracetylquinic Acid, $(OC_2H_3O)_4 \cdot C_6H_7 \cdot COOH$, melts at about 130° — 136° , and is readily soluble in hot water and sodium carbonate, but only sparingly in carbon bisulphide, and almost insoluble in light petroleum.

When quinic acid is heated with acetic anhydride at 170° a mixture of triacetylquinide and tetracetylquinic acid is obtained.

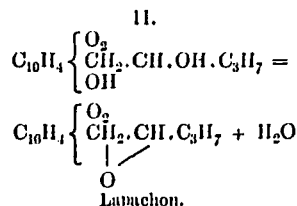
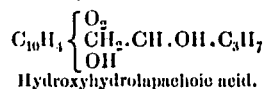
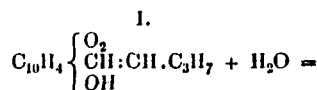
The two triacetylquinides are probably γ - or δ -lactones of quinic acid. Quinic acid seems to contain two OH-groups in the γ - and δ -position to the carboxyl-group for, when an aqueous solution is evaporated with bromine, protocatechuic acid is formed. The formation of meta-chlorobenzoic chloride by heating quinic acid with phosphorus pentachloride also indicates the presence of a γ -OH-group. A third OH-group seems to be in the α -position, as quinic acid, like other α -hydroxycarboxylic acids, evolves carbonic oxide and yields hydroquinonedisulphonic acid when treated with concentrated sulphuric acid.—F. S. K.

Lapacholic Acid and its Derivatives. S. C. Hooker and W. H. Greene. *Ber.* 22, 1723—1727.

The authors have obtained lapacholic acid from a South African wood called *Bethabana*. The substance is identical with that got by Siewert from the lapacho-wood of South America and examined by Paterno. Amongst the derivatives of lapacholic acid obtained by the latter is a body formed by the action of concentrated sulphuric acid, which crystallises in red needles, and to which the name lapachon was given. Paterno assigned the formula $C_{15}H_{11}O_3$ to the acid, regarding it as hydroxyamylene naphthoquinone—



and looked upon lapachon as consisting of two molecules of the acid united by the oxygen atoms of the ketone groups, and as having the formula $C_{20}H_{14}O_6$. The authors have examined this latter body further and find that it possesses all the characteristic properties of a quinone; also its molecular formula as determined by Raoult's method is $C_{15}H_{11}O_3$, and not the double of this. To account for this change it is assumed that lapacholic acid when treated with strong acid first takes up a molecule of water to form an intermediate compound, which is then acted upon by the acid with the elimination of water and the formation of lapachon, which is regarded as a derivative of naphthofurfuran. The following equation expresses the change, using Paterno's formula for the acid:—



Lapachon is not affected by treatment with alkalis or with alkaline carbonates in the cold, but it dissolves in the former on heating, and from this solution acetic acid separates a red oil which solidifies to a yellow crystalline mass melting at 126° C. when pure, and which gave on analysis numbers agreeing with the formula $C_{15}H_{11}O_4$. This is regarded as hydroxyhydrallapacholic acid, the intermediate product in the above reaction. The barium and silver salts of this acid are characteristic.

Other derivatives of lapacholic acid obtained by Paterno are regarded as derived from naphthofurfuran.—C. A. K.

with a lucrative trade, and to supply the medical profession with an abundance of podophyllin, a drug which, as many Anglo-Indians have good reason to know, is a valuable specific in disorders of the liver. Hitherto America has enjoyed a monopoly of the podophyllin plant (*Podophyllum peltatum*), but the researches of Dr. Watt and the analysis by Dr. Hooper, quinologist to the Madras Government, demonstrate that the Himalayan variety (*Podophyllum emodi*) yield three times as much of the valuable resin as the American root, and that it possesses the same medicinal properties. This valuable plant grows wild in the higher, rich, and shady temperate forests from Sikkim to Simla, Kashmir, Hazara Tibet, the Kuram Valley, and Afghanistan. It is fairly plentiful on the northern forest-clad slope of the familiar Shalai hill, seen from Simla; on the almost equally well-known Nagkanda hill and in the Chumba State there are many mixed forests with their glades almost exclusively covered with this peony-rose-like herb. In his notes on the subject Dr. Watt remarks that it is surprising that the natives of India, who have discovered so many drugs, should have failed to detect the properties of the podophyllum root."

XXI.—PHOTOGRAPHIC MATERIALS AND PROCESSES.

Ammonia in the Gelatin Emulsion. Photogr. Wochenbl. 1889, 15, 132.

In preparing an emulsion by the cold process with ammonia, Belitzki recommends that most of the ammonia be neutralised with sulphuric acid towards the end of the process. An excess of acid must be avoided, as the least trace of free acid diminishes the sensitiveness of the emulsion. If some aurin-collodion be placed on a glass plate and the emulsion added to it, a rose-red colour will be produced if the emulsion is alkaline.—E. E. B.

Preparation of Plates Sensitive to Colour. E. Obernetter. Photogr. Wochenbl. 1889, 15, 133.

The emulsion is made with little gelatin and poured over the plates thin. It is not advisable to add the colouring material to the emulsion at once. If a bath be used for dipping the coloured plates, it has the disadvantage of becoming weaker after the immersion of every plate, so it is preferable to pour the colouring material in an extremely dilute solution over each plate separately, after washing it thoroughly. A good recipe for the colour solution is:—50 parts erythrosin solution (1:1,000), 50 silver nitrate solution (1:1,000), 3 ammonia. The plates may be exposed with or without a yellow screen, but in the former case more than twice as long an exposure is necessary. For developing, an alkaline solution of pyrogallie acid or a hydroquinone solution is used.—E. E. B.

Use of Sodium Sulphite in the Fixing-Bath. Photogr. Archiv. 1889, 30, 161.

When a gelatin dry plate, which has been developed by means of pyrogallie acid, is placed in the fixing-bath of sodium hyposulphite, unless the pyrogallie acid solution has been perfectly removed by washing, the plate will become yellow. To obviate this, William Bell recommends the addition of sodium sulphite to the sodium hyposulphite (proportion 1:5). Negatives fixed by this solution have the tone and character of collodion negatives prepared by the wet process, and the bath can be used much longer.

—E. E. B.

Citric Acid in the Hydroquinone Developer. Thomas. Photogr. Archiv. 1889, 30, 162.

The developer consists of two solutions, which are mixed in equal volumes immediately before use. No. 1 consists of 5 parts hydroquinone, 32 sodium sulphite, 2 citric acid, and 1 potassium bromide in 128 parts of water. No. 2 consists of 5 parts caustic soda in 128 of water. This is eight times as powerful as the ordinary pyrogallie acid developer.—E. E. B.

Theory of the Hydroquinone Developer. Phot. Rundschau. 1889, 3, 119.

HYDROQUINONE, $C_6H_6O_2$, is prepared by the action of sulphurous acid on quinone, $C_{10}H_6O_2$. By the action of the former on exposed silver bromide two atoms of hydrogen unite with the bromine liberated and silver is set free, which forms the picture—



Quinone is thus formed again, and the hydrobromic acid combines with the alkali which is necessarily always present in the developer. The alkaline bromide retards development, and on this account old solutions always develop more slowly. The sulphurous acid in the sodium sulphite is supposed to prevent or diminish the rendering of the gelatin brown by the action of the quinone.—E. E. B.

Intensifying by Means of Hydroquinone. Phot. Rundschau, 1889, 3, 194.

If a gelatin negative has been treated with mercuric chloride to intensify it until it has become quite white, instead of then treating it as usual with ammonia it is advised by Dr. Mallmann to wash the plate quite free from any adhering mercuric chloride, and then pour over it a solution of hydroquinone containing sodium sulphite or dip the plate into this solution. The result is an intense negative of a bluish-black tone.—E. E. B.

PATENT.

Improvements in Photometers for Measuring the Intensity of Light in Photographic Camera. J. Decoudum, Paris, France. Eng. Pat. 11,578, August 10, 1888. 8d.

By means of this apparatus the intensity of the light focussed on the screen of the camera, and from that the time of exposure, may be determined. The photometer consists of a series of translucent sectors of a rotary disc, or, as in a modification, of a stationary disc divided into squares or spaces of different degrees of translucency, each bearing a letter or other sign indicating the period of exposure. In the former case the disc is rotated to bring "the gradually darkening luminous points" opposite a lens and eye-piece, while the stationary disc is mounted in a tube also provided with eye-piece and lens. When placed against the focussing screen and viewed through the lens, the letter which is least visible indicates the time of exposure.—O. H.

XXII.—EXPLOSIVES, MATCHES, Etc.

PATENTS.

Improvements in Explosive Compounds. A. V. Newton, London, From A. Nobel, Paris. Eng. Pat. 10,722, July 24, 1888. 4d.

This invention relates to the manufacture of an explosive consisting of nitrate of ammonia and picrate of ammonia in the proportion of three parts of the former to one part of the latter.

Gum arabic, dextrin, or an analogous substance is added in the proportion of about $\frac{1}{2}$ per cent. of the material, with a view of hardening it.—C. N. H.

Improvements in Protecting and Preserving Explosives. C. Lamm, Stockholm, Sweden. Eng. Pat. 11,751, August 15, 1888. 6d.

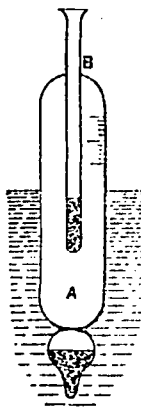
The object of this invention is to protect and preserve explosives which contain hygroscopic salts.

In carrying out the invention, carnauba or other palm wax is employed mixed with Japan wax, naphthalene, paraffin, &c. as a waterproof coating for the explosive when made up into blocks or cartridges.—C. N. H.

XXIII.—ANALYTICAL CHEMISTRY.

On a New Hydrometer. W. Láskn. Zeits. f. Instrumentenkunde, 9, 176.

In consequence of capillary action, measurement by the hydrometer is not certain.



If p be the weight, α a capillary constant, ω the angle of contact (Randwinkel), r the radius of the hydrometer, v the volume immersed, d the density of the liquid, u the volume of the instrument in the air, and δ the specific gravity of the air, then—

$$p + 2\pi r \alpha \cos \omega = v d + u \delta$$

or when $u \delta$ is very small and can be neglected—

$$p + 2\pi r \alpha \cos \omega = v d$$

If, now, p be largely increased, while α and r remain the same, then—

$$p_1 + 2\pi r \alpha \cos \omega = v_1 d$$

$$p_1 - p = (v_1 - v) d; \text{ or } d = \frac{p_1 - p}{v_1 - v}.$$

By means of the instrument shown in the figure which is based on this formula, the influence of capillary action can be avoided. To the upper part of A the tube B is fused. Its use is obvious. The instrument is placed in the liquid and the height read off; a known weight of mercury is then run into B and the height again read off (best by a cathetometer). The difference of height in mm. in weight of mercury in milligrams represents the specific gravity of the liquid.—O. H.

Arsenic in Glycerin (Glycerol). L. Siebold. Pharm. Conference, Newcastle-on-Tyne. Pharm. J. 1889, 205.

The author has investigated colourless and odourless samples of glycerin sold for toilet and pharmaceutical purposes (this Journal, 1889, 639—640). The majority of the samples tested were found to contain arsenious acid varying in proportion from 1 part in 4,000 parts to 1 part

in 6,000. A few samples exceeded this proportion, and one contained as much as 1 part in 2,500. Some samples free from arsenic were traced all to one process of manufacture, described as being that by which, as a rule, glycerin used for dispensing purposes is understood to be used. The process followed in testing, which was demonstrated by the author, was a modification of one recently recommended by Vulpius and Flückiger, which depends upon the action of the gas given off by a mixture of the glycerin with zinc and very dilute hydrochloric acid in a test tube upon a paper cap that has been touched with a drop of a solution of mercuric chloride, a yellow spot being the result of the presence of minute traces of arsenic. A further observation by the author was consistent with arsenic being sometimes introduced into glycerin from the glass of the containing bottle, and it is proposed to further investigate this point; but there was a general agreement among the speakers that such quantities of arsenic as those found in some cases were more probably introduced during the manufacture.—W. S.

Solidification of Nitrogen Trioxide. F. Birlhans. Compt. Rend. 109, 63.

Nitric oxide and oxygen in the proportions of 6—8 volumes of the former to 1 volume of the latter were passed through a U-tube cooled to 54°. The large excess of nitric oxide is necessary to prevent the formation of any nitrogen peroxide. The gases must be well dried previously by passing over pumice stone moistened with sulphuric acid, dry baryta, and lime. The blue liquid which collects in the U-tube may be solidified by a mixture of methyl-chloride and carbonic acid with snow, that is to say, at a temperature of -82°C .

—A. R.

Wiborgh's Volumetric Process for the Estimation of Sulphur. A. Vosmaer. Chem. Zeit. 13, 695.

The author substitutes twill for white calico for the collection of the cadmium sulphide, but otherwise performs the analysis as described by Wiborgh. The standard iron is prepared by fusing cast iron with sulphide of iron. Half a gram of a sample is ample for analysis.—E. E. B.

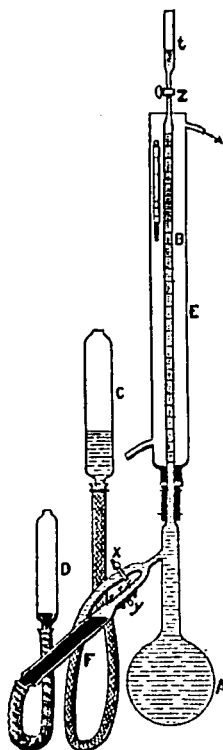
Volumetric Estimation of Sodium Carbonate in the Presence of Sodium Hydrate in Commercial Caustic Soda. H. Goebel. Chem. Zeit. 13, 696.

A drop of an alcoholic solution of phenol phthalein is added to the cold solution and standard acid added till the colour is destroyed; a drop of Porrier's blue (C. L. B.) is added and then more acid till the solution becomes purple (the colour of an ammoniacal solution of copper). The difference between the first and second titration corresponds to half the sodium carbonate present.—E. E. B.

On the Absorption of Atmospheric Gases by Water. O. Pettersson and K. Söndén. Ber. 22, 1439—1446.

The authors have determined the quantity of dissolved gases in water from the Baltic and in the town's water of Stockholm. The results obtained differed so materially from the coefficients of absorption calculated by Bunsen, that it was found advisable to repeat Bunsen's experiments with pure water. The apparatus used is shown in the figure. The side tube of flask A is Y-shaped. One limb is in connexion with the reservoir C, the other with the vessel E, which contains mercury. The enouchone tubing between F and D is surrounded by a coil of copper wire. Before introducing the water, F is entirely filled with mercury and the stopcock y is closed. A and B having been filled with water, x is opened and y closed. All the air is now expelled by vigorous boiling of the water in A and of that portion which has passed into F. The operation takes half an hour. In one experiment N and O may be determined, but not CO_2 .

Sulphuric acid should not be added to the water, otherwise mercurous salt is liable to be formed at the cost of the oxygen expelled. After removing CO_2 with caustic soda, oxygen is absorbed by a solution of pyrogallie acid. The great advantage of this apparatus is that constant and almost perfectly accurate results are obtained with it, as the water to be analysed is shut off from contact with the outer air by mercury. Boiling may be continued indefinitely. It is of the greatest importance that the water used should be kept as nearly as possible at the same temperature while it



is being saturated with air. The authors were only satisfied when no change greater than 1°C. was observable in 5–6 hours.

The following figures were obtained, corresponding to cc. in 1 litre of water, saturated with air, by frequently pouring from one vessel into another:—

Tem- perature of the Water.	Found.			Calculated for Absorption at 760 mm. Pressure.		Per- centage of Oxygen.
$^\circ \text{C.}$	Baro- meter.	N.	O.	N.	O.	$100 \frac{\text{O}}{\text{N} + \text{O}}$
0	772	10.85	10.15	10.63	10.01	38.88
+ 0.00	771	10.58	8.30	10.34	8.28	33.00
+ 6.32	741	10.20	8.18	10.00	8.30	33.55
+ 9.18	720	14.10	7.00	16.68	7.00	33.60
+ 13.70	771	14.37	7.24	14.10	7.14	33.51
– 14.10	770	14.35	7.15	14.16	7.05	33.24

As the relative quantity of oxygen, nitrogen, and carbonic acid contained in a natural water gives important indications concerning its origin and condition as well as to its suitability for drinking purposes, it is desirable that greater

stress should be laid upon the determination of these gases in water analyses than has hitherto been done. Thus, to find a water, abnormally deficient in oxygen, would point to the influence of animal or vegetable life. Attention is drawn to the fact that spring water often contains marsh gas dissolved along with nitrogen. These two gases are obtained together during analysis, and if the volume be larger than is noted down for nitrogen at the given temperature in the above table, the mixture must be further analysed by combustion with oxygen. Thus the authors have found as much as .7 cc. CH_4 per litre in a spring water. Sometimes however there is really an excess of nitrogen, due to the high air-pressure in the regulators of the waterworks, which leads to supersaturation so far as nitrogen is concerned, but not in regard to oxygen.

The town's water was richest in oxygen in September (about 3 cc. over the average); the lowest amount was found in February (about $1\frac{1}{3}$ cc. under the average). The variations in the amount of carbonic acid were just in the reverse order. The maximum of CO_2 was in September (29 cc.) the minimum in March (18 cc.), which facts are probably due to more favourable conditions for oxidation at the summer's heat.—A. R.

The Solubility of Oxygen in Water. L. W. Winkler. Ber. 22, 1764–1774.

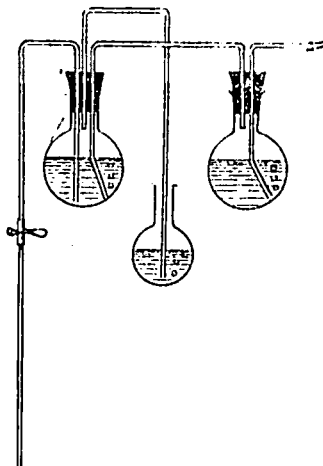
In a former paper (Ber. 21, 2843) the author showed that Bunsen's determination of the absorption coefficients of oxygen, nitrogen, and air required correction. The present paper is devoted to the more accurate determination of the solubility of oxygen in water.

The oxygen was estimated in water saturated with air at different temperatures (0° – 30°C.) by the iodometric method described by the author in a former communication, and from these values the absorption coefficients were calculated.

The analytical method was also improved, regard being had to the fact that under ordinary conditions the reagents themselves contained oxygen in solution. This source of error was overcome by employing almost saturated solutions of the reagents, viz., 80 grms. MnCl_2 , 4 H_2O dissolved and made up to 100 cc. The caustic soda and iodide of potassium were about 12 times normal strength.

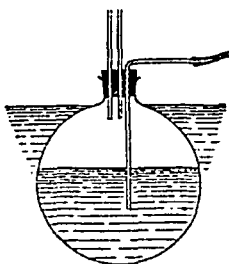
The hydrochloric acid was freed from oxygen by passing carbonic acid through it and then boiling, whereby the carbonic acid was expelled, carrying the oxygen along with it. This process was repeated several times, and the author lays special stress on the necessity of boiling the acid to get rid of the last traces of oxygen. The following apparatus was used: (Fig. 1).

Fig. 1.



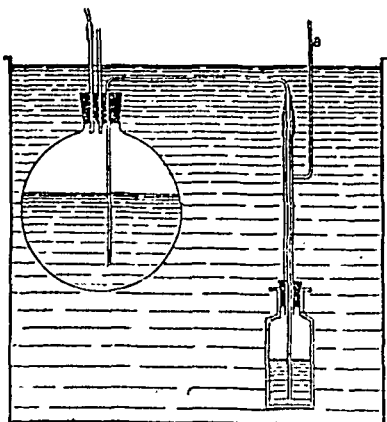
Two boiling flasks of about 100 cc. capacity containing 20 per cent. hydrochloric acid are employed. In one of them the carbonic acid is washed, after which it passes into the second flask. The second flask is provided with a syphon tube for withdrawing the solution of hydrochloric acid after it had been freed from oxygen. The results of the experiments are given, showing that the reagents thus prepared were free from oxygen. Another point is that the amount of oxygen which water is capable of absorbing from the air is influenced by the height of the column of water. Consequently it is best to calculate the solubility of gases in terms of a column whose height is = 0. It suffices to add half the pressure of the water column to the barometer reading and take that as the atmospheric pressure. The estimations were made as follows: A 4-litre flask (Fig. 2)

Fig. 2.



was half filled with carefully distilled water, and the neck closed with an india-rubber stopper through which passed two short straight tubes and one long bent one. Thoroughly purified air was led through the long tube and the contents of the flask shaken from time to time. The flask was kept at constant temperatures. When the water was thoroughly saturated with air the height of the barometer was noted, and by means of the arrangement shown in Fig. 3, some

Fig. 3.



of the water was carefully transferred to a carefully calibrated flask, one of the short tubes being meantime connected with the gas-holder supplying the pure air. The calibrated flask was taken out of the water-bath and the temperature of its contents carefully determined, the necessary reagents were then added, and the whole thoroughly mixed. The titration took place after 24 hours.

During this time the flask was closed against air entering by placing its stopper downwards in a beaker full of water. Detailed results of the determinations are given, and from

these have been calculated the solubility and absorption coefficient of oxygen. "That quantity of a gas reduced to 0° and 760 mm. which is dissolved by unit volume of a liquid at 760 mm. is the *solubility* (β') of the gas. On the other hand, the quantity which is taken up by a liquid when the pressure of the gas itself, without the tension of the liquid, amounts to 760 mm. is called the *absorption coefficient* (β) of the gas. In both definitions a column of liquid height = 0 is understood."

In the following table the results of the author's determination of the *absorption coefficient* (β') and the solubility of oxygen in water under normal pressure are compared with Bunsen's figures, and the last column (β' 209°6) gives the quantity of oxygen in cc. in 1,000 cc. of water saturated with air.

t degree C.	Bunsen's Absorption Coefficient.	β .	β' .	β' 209°6
0	0.01114	0.01890	0.01880	10.187
1	0.01007	0.01759	0.01728	9.910
2	0.00907	0.01633	0.01601	9.643
3	0.00810	0.01512	0.01479	9.387
4	0.00717	0.01397	0.01362	9.142
5	0.00628	0.01286	0.01250	8.907
6	0.00544	0.01181	0.01142	8.682
7	0.00465	0.01080	0.01040	8.467
8	0.00389	0.00991	0.00941	8.260
9	0.00317	0.00891	0.00841	8.063
10	0.00250	0.00802	0.00756	7.873
11	0.00189	0.00718	0.00670	7.692
12	0.00133	0.00637	0.00587	7.518
13	0.00082	0.00560	0.00507	7.352
14	0.00034	0.00486	0.00431	7.192
15	0.00000	0.00415	0.00358	7.039
16	0.00019	0.00347	0.00288	6.891
17	0.00014	0.00283	0.00220	6.750
18	0.00004	0.00220	0.00155	6.614
19	0.00003	0.00161	0.00093	6.482
20	0.00000	0.00103	0.00032	6.356
21	..	0.00048	0.00007	6.233
22	..	0.00004	0.00000	6.114
23	..	0.00000	0.00000	6.000
24	..	0.00000	0.00000	5.880
25	..	0.00000	0.00000	5.770
26	..	0.00000	0.00000	5.660
27	..	0.00000	0.00000	5.554
28	..	0.00000	0.00000	5.450
29	..	0.00000	0.00000	5.357
30	..	0.00000	0.00000	5.265

—C. N. H.

Proceedings of the Austrian Society of Chemical Industry.
Meeting of June 1, 1889. Chem. Zeit. 13, 781.

J. Stoklasa drew attention to the solubility in water of crystallised calcium tetrahydrogen phosphate. According to the volume of water employed, the salt is more or less

decomposed into monohydrogen calcium phosphate and phosphoric acid, as shown below :—

Ratio of Salt to Water used for Solution.	Percentage of Salt Decomposed.
1 : 1	25
1 : 25	12.5
1 : 50	6
1 : 50	3
1 : 200	0.1

One part of calcium tetrahydrogen phosphate, therefore, requires 200 parts of water for complete solution. The liberated phosphoric acid was estimated by titration with decinormal soda and methyl orange, the total amount of phosphoric acid being determined with molybdic acid. The quantity of undecomposed phosphate in the filtrate from the monohydrogen calcium phosphate was calculated from the amount of calcium found.

The same chemist further mentioned a method of determining phosphoric acid in superphosphates, &c. 10 grms. of the superphosphate are brought into a litre flask and the flask filled to the mark. The solution is filtered, and 50 cc. of the filtrate are treated with ammonium citrate and magnesia mixture and shaken up for 5–6 minutes; then a volume of ammonia equal to one-fourth the volume of the mixture is added. The precipitated magnesium ammonium phosphate is collected, ignited, and weighed as pyrophosphate. The ammonium citrate solution is made by dissolving 250 grms. of citric acid in 500 cc. of water; 20 cc. of this solution, after neutralisation with ammonia, are used for the test. The following results show the accuracy of the method :—

Material Analysed.	Percentage of Phosphoric Acid.		
	Found by Ammonium Citrate Method.	Found by Molybdic Acid Method.	Calculated.
Monohydrogen calcium phosphate, (pure)	52.36	52.16	52.2
Tricalcium phosphate $[Ca_3(PO_4)_2, 2H_2O]$...	41.30	41.27	41.05
Bone meal	26.40	26.60	..

—E. B.

On Schützenberger's Process for the Estimation of Dissolved Oxygen in Water. Sir H. E. Rosecoe and J. Lunt. J. Chem. Soc.

The authors have lately made the above method the subject of careful investigation, and have succeeded for the first time in placing this important estimation on a satisfactory footing.

As the measurement of the amount of dissolved oxygen in waters, whether pure or impure, has an important bearing on many technical processes, it may not be unfitting to give a short account of this investigation in the Journal of the Society of Chemical Industry.

The method depends on the reducing action of sodium hyposulphite, Na_2SO_3 ,* on a blue solution of indigo-carmin. A dilute solution of hyposulphite added to the blue liquid turns it yellow; in this condition the liquid is a most delicate test for the presence of free oxygen, either gaseous or dissolved.

The process hitherto used may be given shortly as follows :—A large Woulff's bottle is filled with hydrogen, and about 30 cc. of water introduced into the vessel. To this liquid is added indigo-carmin sufficient to colour it

blue. Hyposulphite solution is now added from a burette until the blue is turned to yellow. The water to be examined is now added to the reduced solution, with an immediate restoration of the blue colour, due to oxidation by the dissolved oxygen contained in the added water. The amount of oxygen is estimated by noting the volume of hyposulphite required to restore the yellow colour, or in other words, that required to counteract the oxidation produced by the amount of oxygen added. Two curious observations had previously been made by Schützenberger and confirmed by other observers.† One was that after the estimation was effected as above described, the reduced yellow liquid quickly regained its former blue colour, and this, if again decolourised, repeatedly returned to blue until about double the previous amount of hyposulphite had been used.

The other observation was, that if a much larger quantity of indigo-carmin be employed, the amount of dissolved oxygen found is about double that obtained when only a small amount of indigo is used, Schützenberger's explanation being that the reaction between hyposulphite and dissolved oxygen is a different one to that taking place between hyposulphite and indigo-carmin; that in the former case half the oxygen became latent as hydroxyl, with subsequent slow liberation of oxygen, whilst in the latter reaction no hydroxyl was formed. Hence, in the first case, only half the total oxygen is obtained, whilst in the second the whole is found.

The authors explain these observations as follows :—

Low Result when the Water has been exposed to Hydrogen.—The cause of these anomalous results was indicated by an experiment in which the water to be analysed, contained in a Winchester quart bottle, was allowed to stand in an atmosphere of hydrogen overnight, estimations being made before and after such exposure. The results obtained show that diffusion is the disturbing influence, for when aerated water is introduced into an atmosphere of pure hydrogen it immediately begins to lose oxygen by diffusion into the hydrogen until an equilibrium is established. This is a fact hitherto overlooked by the experimenters with this process.

Explanation of the previous Experiments.—This explains the previous observations, viz., that the highest results are obtained when the hyposulphite is added quickly and immediately after the introduction of the water into an atmosphere of hydrogen, because the oxygen is estimated and combined before it has had time to diffuse to any great extent, the slight difference in the time of exposure between a quick and a slow titration being quite sufficient to give a marked loss of oxygen by diffusion during the slower addition of hyposulphite. The variation in the results of the apparently identical experiments first given, being explained by the slightly longer or shorter time taken over each titration, and the consequent greater or less facility for diffusion.

Results less in proportion to the amount of exposure to Hydrogen.—This conclusion, that diffusion is the disturbing influence, is borne out by further experiments easily suggested by the preceding.

Conditions influencing the Amount of Diffusion.—The rapidity and amount of the diffusion of dissolved oxygen depend on (1) the surface area of water exposed to hydrogen, influenced by the size and shape of the vessel and the degree of agitation during titration. (2) The volume of the atmosphere of hydrogen. (3) The time of exposure, up to the point of equilibrium (under the law of partial pressures). (4) The volume of water.

The authors show that the large size of the vessel hitherto recommended for the estimation is greatly conducive to the rapid loss of oxygen by diffusion, and by using the same solutions in a smaller titration vessel a greatly increased amount of oxygen was obtained.

Reason for the Return of the Blue Colour.—In carrying out the oxygen estimation in the usual manner, the blue colour after having once been destroyed by the hyposulphite almost immediately returns, and if this subsequent colouration be destroyed the colour will again return, and so on

* Prepared by the action of zinc dust on a strong solution of sodium bisulphite saturated with sulphur dioxide, with subsequent neutralisation by lime.

† Dupré, *The Analyst*, 10, 160; and Ramsay and Williams *Chem. Soc. J. (Trans.)* 49, 761.

for a long time. The blue colour returns even when the hydrogen used is perfectly freed from all traces of oxygen by passing through the two long tubes filled with beads moistened with the alkaline solution of potassium pyrogallate used in all our experiments.

No Return of the Blue Colour when Free Oxygen is Absent.—If the liquid containing indigo-carminine and also the atmosphere above it are perfectly free from uncombined oxygen, and hyposulphite be added until the blue is turned yellow, no return of the blue colour takes place.

Proof of the Diffusion of Dissolved Oxygen into Hydrogen.—A positive proof that when pure hydrogen comes in contact with aerated water it is contaminated with oxygen, has been obtained as follows:—

A large flask was filled with a fairly strong indigo-carminine solution which was decolourised carefully with hyposulphite. Matters were so arranged that pure hydrogen, freed from all traces of oxygen as before stated, could be passed through aerated water, and thence into the large flask, driving out the sensitive yellow liquid through a tube passing to the bottom of the flask. In this way the hydrogen, contaminated with oxygen from the aerated water, is imprisoned above a solution which will indicate the presence of oxygen by the formation of a blue colour. This blue colour *was* observed at the surface of the liquid, and after a few minutes it became very intense and unmistakable.

Influence of the Amount of Indigo-Carminine used.—These experiments explain a singular observation made by Schützenberger, namely, that by using a small amount of indigo-carminine only half the dissolved oxygen is estimated,

when the first decolourisation of the solution is effected; whilst when a much larger quantity of indigo-carminine is employed *the whole* of the dissolved oxygen is found. The explanation given by him being that the reaction between reduced indigo and dissolved oxygen is a different one from that taking place between hyposulphite and dissolved oxygen, inasmuch as in the latter reaction half the oxygen becomes latent as hydroxyl with subsequent slow liberation of oxygen, whilst in the former no hydroxyl is formed.

His observation, however, will bear another interpretation, namely, if more reduced indigo be present than is sufficient to combine with all the dissolved oxygen contained in the added water, this oxygen is immediately combined by the reduced indigo and its diffusion prevented. Hence *all* the oxygen is obtained. The use of so large a quantity of indigo, however, so disturbs the end-reaction, from the presence of so much of the yellow compound, that "it is difficult to fix the point at which the last trace of blue has been discharged with any degree of accuracy." (Dupré.)

A new method must satisfy the following conditions:—

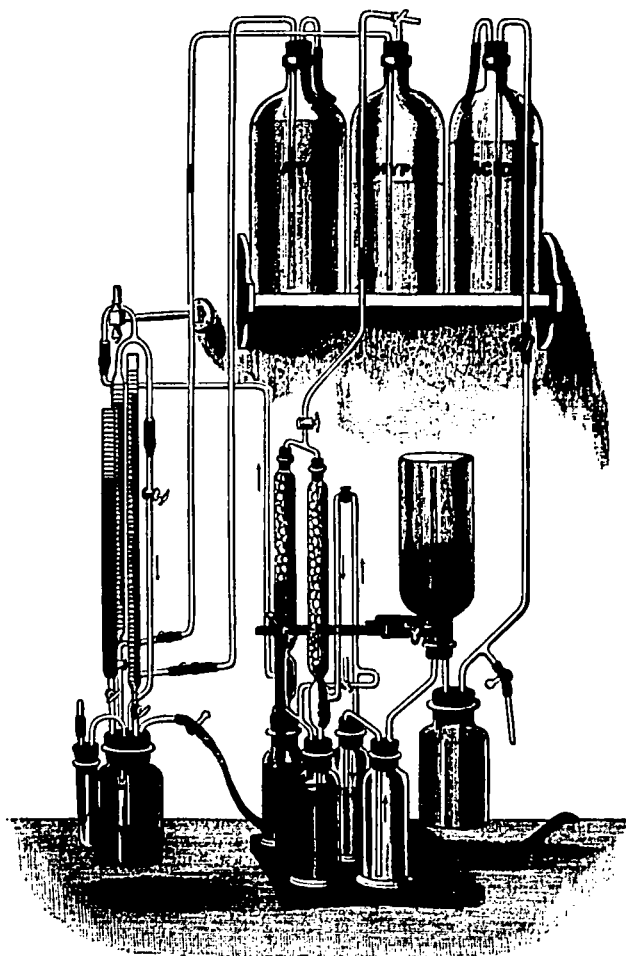
(1.) The aerated water must not at any time expose its *uncombined* oxygen to an atmosphere of hydrogen.

(2.) Precautions must be taken to ensure the use of an atmosphere perfectly free from oxygen, and the apparatus used must not permit any ingress of air.

(3.) When the vessel in which the analysis is made is first filled with water to expel air or to obtain the sensitive yellow liquid, the first portion of gas, having been in contact with aerated water, is contaminated with oxygen and must be entirely driven out.

Apparatus adopted.—Fig. 1 shows the apparatus adopted by us for carrying out the estimation.

Fig. 1.



It consists essentially (1) of an apparatus for the continuous generation and purification of hydrogen, by the action of dilute sulphuric acid on zinc; (2) a 200 cc. wide-mouthed bottle fitted with three burettes with glass taps, inlet and outlet tubes for a current of hydrogen, and an outlet tube for the titrated liquid; (3) Winchester stock-bottles of hyposulphite, indigo (not shown), and water, communicating with their respective burettes by glass syphons.

The hydrogen generated in A passes through two wash-bottles containing caustic potash, thence through two Emmerling's tubes filled with glass beads moistened with an alkaline solution of potassium pyrogallate, an arrangement being made whereby the beads may be re-moistened with fresh pyrogallate from the bottles beneath, the liquid being forced up by hydrogen pressure.

Pure hydrogen is supplied continuously (1) to the stock-bottle of hyposulphite, (2) to the hyposulphite burette, and (3) to the titration bottle.

The New Method.—A sample of water is examined as follows:—

(1.) 20 cc. of the water is introduced into the small bottle and about 3 cc. of indigo solution added.

(2.) A moderate current of hydrogen is passed through the blue liquid by a very fine jet for three minutes to free both water and supernatant gas from free oxygen.

(3.) Hyposulphite is now carefully added, during the flow of hydrogen, until the change from blue to yellow occurs, taking care not to overstep this point.

(4.) A further measured quantity of hyposulphite is now added (say 10 cc.) sufficient to combine with all the dissolved oxygen in the volume of water, 50–100 cc., proposed to be used in the estimation.

(5.) The important point now is, that the water is run in from a burette by a capillary tube passing *beneath the surface of the liquid* to the bottom of the vessel. Thus we introduce the water into a liquid which will *at once* combine the free oxygen, and thus prevent its diffusion on coming in contact with the hydrogen, the reduced indigo acting as an indicator for the complete oxidation of the hyposulphite. The liquid is kept in constant motion during the addition of the water, which is shut off the moment a permanent blue colour appears.

(6.) The blue is decolourised by a further slight addition of hyposulphite. The volume of water used, and the total hyposulphite, minus the first addition, are noted, and the estimation repeated for confirmation.

When the water contains very little oxygen the second addition of hyposulphite may be omitted, the reduced indigo-carmine being sufficient to take up all the dissolved oxygen. In this case, care must be taken that the oxygen added should require not more than half the hyposulphite first added to decolourise the indigo-carmine.

Standardisation of the Hyposulphite.—In order to complete the estimation it is necessary to know the strength of the hyposulphite solution employed. This has entailed a very considerable amount of labour, inasmuch as of the two methods hitherto adopted for this purpose, the first was found to be fundamentally wrong, and the second to depend on erroneous data.

The authors examine the copper method of standardisation, depending on the decolourising effect of hyposulphite on a standard solution of ammonio-cupric sulphate.

They adopt the method of standardisation depending on a titrating of fully aerated distilled water, aerated at a known temperature and pressure, but find, on investigation, that the oxygen value of such water is inaccurately given by Bunsen's coefficients of absorption, previously used for the standardisation, and they substitute the following values which they have determined by using apparatus specially constructed for the purpose of extracting the dissolved gases by boiling the water *in vacuo*, and subsequently measuring the volume of oxygen.*

Method of Aeration.—Before giving the results of the estimations made with distilled water saturated with air, it is necessary to describe the method of aeration.

Freshly distilled water adjusted to the required temperature was aerated by vigorous agitation in two Winchester

quart bottles half full of water, the air being renewed five or six times by emptying the contents of one bottle into the other and again dividing into two portions, which were repeatedly agitated with fresh air.

When aerated, one bottle was filled and allowed to stand, stoppered, for half an hour to an hour, to get rid of minute air-bubbles. Then the gases were extracted and analysed, the barometer having been read to obtain the pressure at which the water was aerated.

The numbers obtained indicate close agreement between the several experiments at each temperature, and also throughout in the ratio between the oxygen and total oxygen plus nitrogen, a ratio always between 33 and 34 per cent.

They show a wide discrepancy from Bunsen's numbers, but agree well with Dittmar's determinations.†

The following table has been constructed, giving the oxygen values for fully aerated distilled water for each half degree between 5° C. and 30° C., and for an *observed*‡ pressure of 760 mm.:—

OXYGEN DISSOLVED BY DISTILLED WATER. 5°–30° C.

Temp. C.	Cc. Oxygen N.T.P. per Litre Aq.	Diff. for 0.5° C.	Temp. C.	Cc. Oxygen N.T.P. per Litre Aq.	Diff. for 0.5° C.
5.0	8.68		18.0	6.61	
5.5	8.58	0.10	18.5	6.47	0.07
6.0	8.49	0.09	19.0	6.40	0.07
6.5	8.40	0.09	19.5	6.34	0.06
7.0	8.31	0.09	20.0	6.28	0.06
7.5	8.22	0.09	20.5	6.22	0.06
8.0	8.13	0.09	21.0	6.16	0.06
8.5	8.04	0.09	21.5	6.10	0.06
9.0	7.95	0.09	22.0	6.04	0.06
9.5	7.86	0.09	22.5	5.99	0.05
10.0	7.77	0.08	23.0	5.94	0.05
10.5	7.68	0.08	23.5	5.89	0.05
11.0	7.60	0.08	24.0	5.84	0.04
11.5	7.52	0.08	24.5	5.80	0.04
12.0	7.44	0.08	25.0	5.76	0.04
12.5	7.36	0.08	25.5	5.72	0.04
13.0	7.28	0.08	26.0	5.68	0.04
13.5	7.20	0.08	26.5	5.64	0.04
14.0	7.12	0.08	27.0	5.60	0.04
14.5	7.04	0.08	27.5	5.57	0.03
15.0	6.96	0.07	28.0	5.54	0.03
15.5	6.88	0.07	28.5	5.51	0.03
16.0	6.82	0.07	29.0	5.48	0.03
16.5	6.75	0.07	29.5	5.45	0.03
17.0	6.68	0.07	30.0	5.43	0.02
17.5	6.61				

Indigo Standardisation.—When once the hyposulphite has been carefully standardised by distilled water, this rather troublesome aeration may be avoided by finding the oxygen-value of the indigo-carmin solution. This solution remaining constant may be used for the subsequent standardisation of the hyposulphite.

† "Challenger" Reports, Vol. I., Pt. I., p. 100.

‡ In this table the results are calculated for aeration at an *observed* barometric pressure of 760 mm. When the observed pressure is *below* 760 mm., the value must be *subtracted* for every 10 mm. diff. The same value must be *added* when the pressure is *above* 760 mm.

It is only necessary to take a suitable quantity of indigo solution, diluted with water if necessary, free it from all dissolved oxygen by a current of pure hydrogen continued for five minutes, they carefully decolourise with hyposulphite, the value of which has been carefully found by using aerated distilled water. The hyposulphite solution slowly loses strength even when kept in hydrogen, and should be standardised daily when required to be used.

Comparison of the New Method with the Volume of Gas found.—We next proceed to apply the volumetric process as thus modified to the examination of samples of water from different sources, and to compare the results with the absolute volume of gas obtained by boiling out the same water *in vacuo*.

A further comparison was made with very impure Thames water collected at Crossness, opposite the southern outfall for London sewage, 12 miles below London Bridge.

These in like manner indicate that the method gives good results, even with an impure water of this character.

The process thus applied to waters of such varied character, and containing such widely different amounts of oxygen, with a mean difference of only 0.5 cc. from the value obtained by extracting and measuring the gases, shows that, although perfect accuracy cannot be guaranteed for the process thus improved, yet the results obtained are not far removed from the truth, and certainly give very valuable indications as to the oxymetric condition of samples of water submitted to its operation.

Influence of Acidity and Alkalinity.—Although the method is applicable to pure and impure waters, and even to brackish waters, this is not the case if the water contains even small quantities either of free acid or alkali. Thus, if $\frac{1}{1000}$ th part of sulphuric acid be added to pure water, not only is the oxygen value obtained a different one, but the whole reaction is disturbed. The volume of hyposulphite required for the first decolourisation is greater; this only produces a light straw-yellow, instead of the fine deep yellow of a normal titration. The delicacy of the reaction is altogether destroyed, a much larger volume of water than usual only slowly producing a light blue colour, instead of the distinct colouration produced by even half a cubic centimetre of pure water in a normal titration. Oxalic acid has a similar action. The same proportion of ammonia has an entirely opposite effect. The oxygen value is raised instead of lowered, and the delicacy of the reaction is not impaired, although the colour reaction is very much modified.

Sodium bicarbonate has no effect.

Of course, when other substances than oxygen, which decompose hyposulphite, are present in the water, the accuracy of the method is proportionately disturbed.

Delicacy of the Reaction.—With pure water, the change of colour is a very highly sensitive reaction and the most delicate test for the presence of free oxygen we possess, one part of oxygen in two million parts of water being sufficient to bring about a distinct change of colour.

Diffusion of Oxygen through India-rubber.—The delicacy of this reaction may be employed to show the rapid diffusion of atmospheric air through india-rubber tubing. The experiment is made as follows:—

100 cc. of water and 3 cc. of indigo-carmin are freed from oxygen and decolourised in the usual way. The liquid is then forced out by hydrogen pressure on opening A.

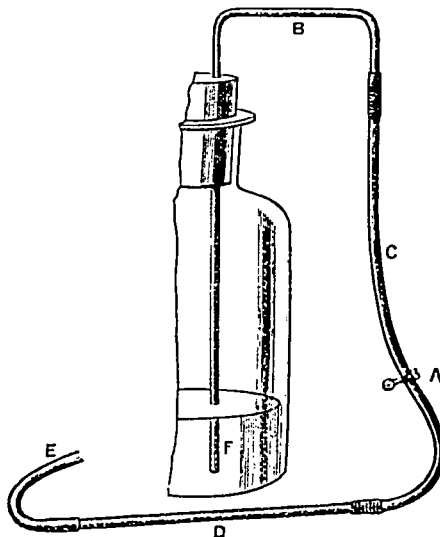
When the liquid has flowed through B, C, D, E, for a few seconds, the pinch-cock A is closed.

The liquid is seen to be yellow at F, B, and D, and is allowed to remain in the india-rubber tube C for five minutes. If now A be carefully opened, so as to allow the former contents of C to be seen in the glass tube D, it will at once be observed that whilst the liquid in F, B, and D had remained unchanged, that in C had turned quite blue, from the absorption of atmospheric oxygen diffused through the india-rubber. Even one minute's exposure is sufficient to give an indication.

This experiment has a practical bearing on the construction of the apparatus as figured in Dupré's paper and in Sutton's work on volumetric analysis, 5th edition, in which the hyposulphite solution is made to pass through several

feet of india-rubber tubing before reaching the titration vessel, its strength, of course, diminishing with the time of remaining in contact with the india-rubber.

Fig. 2.



In the form adopted by the authors, the hyposulphite is conveyed from the stock-bottle to the titration vessel by a glass syphon, and only sufficient india-rubber used to accommodate a pinch-cock and to make one other connexion necessary for the free motion of the titration vessel.

Suggested Applications of the Method.—This process is perhaps most applicable to water analysis. It is well known that organic and bacterial contamination most frequently exist side by side, the one calling forth the other. The activity of these micro-organisms shows itself in the diminution of the dissolved oxygen, which is used up by the bacterial forms during their active life in changing the organic impurities present in the water. Then, again, the process might be used in the brewery, more especially at the fermentation stage of the wort. The deleterious effect of a sluggish fermentation is often felt by the brewer, and this may be due in part to the low state of aëration of the wort.—J. L.

Estimation of Ferrocyanogen in Spent Material from Gas Purification. O. Knublauch. Jour. f. Gasbeleuchtung, 1889, 450—459 and 493—500.

The author points out that the difficulties surrounding the estimation of ferrocyanides in spent gas purification material arise from the impurities present, and he asserts that neither titration with copper sulphate nor with permanganate give reliable results, inasmuch as, in the latter case, the material in question always contains substances other than ferrocyanides which are readily oxidised by permanganate, even after the removal of thiocyanates. Whilst in the former case the impurities interfere either by directly reacting with the copper sulphate or by impairing the delicacy of the end reaction. Moreover, neither the amount of iron nor cyanogen present can be regarded as an accurate measure of ferrocyanides; for according to our present knowledge of their composition it cannot be stated with certainty that these constituents are really present in the form we assume them to be—in fact, the author has come across compounds which resemble, but which are not identical with ferrocyanides. Attention is drawn to the difficulty of removing all the iron not in the form of ferrocyanide in the presence of the different kinds of organic matter present; the author has also observed the decomposition of ferrocyanides with the deposition of iron hydroxide. In both these cases the iron numbers would give incorrect results. In fact, the author has come to the conclusion that

there is no method by which the commercial value of the spent material can be accurately gauged, and therefore he suggests that a method ought to be adopted which will give some insight into the technical value of the material, and involve processes similar to those used in its technical application. And with this object in view, he has devised a method on the following scheme:—Conversion of the insoluble ferrocyanogen into soluble ferrocyanides, purification of the extract, and then estimating the ferrocyanide direct by titration with copper sulphate. Having ascertained, in a series of special experiments, the temperature at which the drying might be conducted without decomposing the material; the conditions under which the conversion of insoluble into soluble ferrocyanides might be effected with the smallest production of substances which would interfere with the titration, such as the quantity and strength of the potash duration of, and temperature during contact; best means of removing interfering substances; modes and accuracy of testing—after completing these investigations, the author incorporated the results in, and adopted the following method:—The whole sample of 200–250 grms. is dried at 50°–60° C. until of constant weight, taking from 5 to 6 hours. The dried sample is pulverised, and passed through a sieve with 360 meshes to the square centimetre; if any sawdust is present it remains on the sieve, and is weighed separately. 10 grms. of the sifted stuff is treated in the cold with 50 cc. of a 10 per cent. solution of potassium hydroxide in a flask marked at 250 cc., and at 255 cc., either for 15 hours with frequent agitation, or for 16 hours agitating only during the first and last two hours, the flask is then filled to the 255 cc. mark, and the contents filtered. 100 cc. of the filtrate is run into a hot solution of ferric chloride, containing 60 grms. of ferric chloride, and 200 cc. of hydrochloric acid (sp. gr. 1.19) per litre, and the whole filtered hot (at about 80° C.) through a folded filter covered over in a hot water funnel, the precipitate is washed with hot water, but not for too long a time. The filter is spread in a porcelain dish, and treated with 20 cc. of 10 per cent. potash, taking care that none of the Prussian blue escapes decomposition; the liquid is washed into a quarter litre flask, and tested for hydrogen sulphide with sodium nitroprusside. If there is not an immediate and distinct reaction, the liquid is filtered, 50 to 100 cc. acidified with 2.5 or 5 cc. of sulphuric acid (1:5), and titrated with copper sulphate. If hydrogen sulphide is present, the unfiltered liquid is treated with 1 to 2 grms. of lead carbonate, filtered quite clear, and then titrated. The copper sulphate solution contains 12 to 13 grms. per litre, and is standardised by titrating with a solution of pure potassium ferrocyanide containing 4 grms. per litre. To test the completion of the reaction either the spot or filter test is employed; in the former a drop of the liquid is placed on filter-paper, and a drop of ferric chloride is placed near it; then, as the drops spread and come in contact, the appearance of a blue colouration where the liquids meet indicates incomplete reaction; but where this formation of the blue ceases to be observed the action is completed according to this test. In the filter test a portion of the liquid is carefully filtered through a minute filter, and the clear liquid tested with ferric chloride; this is the more delicate test, and therefore always registers slightly higher results than the spot test. Care must be taken to always use the same proportion of acid in the titration, and to allow, in all cases, the same time for the copper sulphate to react, and also for the appearance of the blue colouration in testing: 2 minutes for the spot test, and $\frac{1}{2}$ to $\frac{3}{4}$ minute for the filter test. Calculations may be based on either the spot or filter litre except when lead has been used, then the latter only must be used for the calculations, which are simple enough. Multiplying by 0.511 converts percentage of potassium ferrocyanide into hydroferrocyanic acid; by 0.678 into Prussian blue; by 0.369 into cyanogen; by 0.383 into hydrocyanic acid.

As a rule the spot and filter titrations only differ from 0.2 to 0.6 cc. when from 8 to 12 cc. of the copper solution of the above strength is used, and the addition of the last $\frac{1}{10}$ cc. of copper solution is sufficient to dispel the blue colouration which gives way to a brownish hue or to a colourless state. Another point is that most filter-papers contain sufficient

iron to give the blue colouration without any further addition of iron, and with pure ferrocyanides and most samples of spent material from gas purification, the titrations obtained by the spot test with and without the addition of iron are alike. But the author observed that sometimes instead of the blue colour giving way readily in the usual manner, the solution in the filter test remained persistently green or greenish-yellow, the consequence being that the filter test registered much higher than the spot test; moreover in such cases the spot test with iron registered higher than the spot test without iron. A number of experiments were instituted to inquire into this matter, and it appears to be due to those substances already referred to, which apparently contain iron and cyanogen in varying quantities; they also contain sulphur. They appear to undergo decomposition and gradually yield ferrocyanides. They are dissolved by potash, and are apparently precipitated by ferric chloride, but are not, it seems, of the same value as ferrocyanides, but as they undergo conversion they ought to be estimated. In fact, on keeping for some time they disappear, and material containing them will after some months cease to show indications of them, and the spot and filter titrations will be normal; so also material which has been stored or regenerated does not contain them. The author suggests, as the outcome of his experiments, the following method of correcting for these impediments:—Add to 200 or 300 cc. of the solution slightly more copper sulphate solution than is required by the spot titration, too little copper sulphate causes decomposition even with pure ferrocyanide; filter, pour the filtrate into hot ferric chloride, decompose and titrate the Prussian blue formed, and, if it is thought desirable, the filtrate from this precipitate is treated in the same manner. The numbers obtained in this way are added as corrections to the numbers obtained in the usual manner, and it is then found that the spot and filter titrations approximate more and more, the latter becoming less the former increasing. It therefore seems that in these cases the spot test shows too little and the filter test too much, and therefore an alternative for the fractional titration would be an agreement between the parties concerned to adopt some mean result for the basis of valuation; for instance, mean of the spot test without iron and the filter test. Tables of analytical results of the examination of some purification materials are given. It is pointed out that in valuing material the ferrocyanogen as well as the hydrogen sulphide absorbing power must be considered.

The Separation of Copper from Antimony. Finkener. Mitth. Königl. techn. Versuchs. 1889, 76–86.

The separation is effected in two stages: first by the precipitation of the bulk of the copper, as cuprous iodide in an acid solution, and then by the removal of the remainder as sulphide in an ammoniacal solution. The author has established the following facts, serving as a basis for his method:—

- (1.) Cuprous iodide carries down antimony even in the presence of tartaric acid from a nitric acid solution of the two metals, but does not do so if the antimony be first converted into a double salt of antimony pentafluoride and an alkaline fluoride by the addition of the latter.
- (2.) Free iodine in the presence of an alkaline fluoride converts antimony trifluoride into the pentafluoride, though somewhat slowly; the process is hastened by heat and the addition of sulphuric acid.
- (3.) Sulphurous acid reduces antimony pentafluoride slowly in the presence of potassium iodide; the addition of hydrochloric acid hastens the reaction, but the presence of potassium fluoride almost completely inhibits it.

From this the following method is derived:—If the solution to be treated contain the antimony as a pentad compound all that is necessary is to add an alkaline fluoride, then potassium iodide and sulphurous acid, heat gently, filter off the bulk of the copper as cuprous iodide, and wash with boiling water made acid with sulphuric acid. In the event of the antimony being in the triad state the

solution may be first treated with chlorine or bromine water, or oxidation may even be effected by the iodine liberated by the action of the cupric salt also present on the potassium iodide, provided time be allowed for it before the addition of sulphurous acid.

The filtrate containing all the antimony and a little copper is oxidised by bromine water, and the metals precipitated together by sulphuretted hydrogen, the mixed sulphides dissolved in hydrochloric acid with the addition of potassium chlorate, ammonium tartrate added, and ammonia in excess; the resulting solution is heated on a water-bath and sulphuretted hydrogen water added little by little until no further precipitation occurs. By thus minimising the quantity of ammonium sulphide present, re-solution of cupric sulphide is avoided.

The filtrate containing only the antimony is acidulated with sulphuric acid and sulphuretted hydrogen passed through; the antimony is determined in the sulphide thus precipitated by heating it, together with its filter, with hydrochloric acid, and determining the sulphuretted hydrogen evolved by standard iodine solution. The author has established that antimony sulphide precipitated in the presence of sufficient ammonium tartrate to convert all chlorine present into ammonium chloride is of normal composition, and yields the theoretical quantity of sulphuretted hydrogen when heated with hydrochloric acid. (Rose, *Analyt. Chem.* 6th edit., II., 478.)

The analytical results quoted show the method to be reliable, if potassium or sodium fluoride in the proportion of about 1 grm. to 0.275 grm. of antimony be used, and the solution in which the first stage of the separation is effected be about 200 cc.

The process is also stated to be equally applicable to the separation of copper and arsenic.—B. B.

Analysis of Crude Tartrates. N. v. Lorenz. *Chem. Zeit.* **13**, 693—694.

THE author defends the statements made by him in a former criticism upon Goldenberg's original process for analysing tartrates (this Journal, 1888, 136 and 239).

—E. E. B.

On the Sulphate and Carbonate of Lithium. F. Kobbé. *Pharm. Zeit.* **34**, 312.

IN most text-books it is stated that lithium sulphate is soluble in alcohol. This, according to the author, is not correct, as lithium sulphate is insoluble in strong alcohol, though it is taken up, according to the quantity of water present, by dilute alcohol. On this account the following method adopted by the German Pharmacopœia for testing the freedom of lithium carbonate from potassium, sodium and magnesium salts is not satisfactory:—"0.1 grm. of lithium carbonate dissolved in a few drops of dilute sulphuric acid must yield a clear solution on addition of 4 grms. of alcohol." Under these conditions even the purest carbonate gives a marked precipitate, on account of the insolubility of the lithium sulphate. The author suggests the following:—

A solution of 0.5 grm. of lithium carbonate in 10 grms. of dilute sulphuric acid must remain clear on addition of 20 cc. of alcohol; 3 per cent. of potassium carbonate or 0.5 per cent. of calcium carbonate produces a turbidity.

—O. H.

The Presence of Pyridine in Amyl Alcohol; a New Compound of Picric Acid. H. von Asbóth. *Chem. Zeit.* **13**, 871—872.

A NUMBER of specimens of amyl alcohol were found to yield, when used as a solvent for picric acid, crystals of what proved to be pyridine picrate, thus pointing to the presence of pyridine in the samples tested, none of which had been denaturated. Hungarian amyl alcohol was found to be free from pyridine, and this is considered by the

author to be due to the fact that in Hungary the spirit is distilled by steam (Bohm-Fredersdorf or Ilges' method is employed), whilst in many parts of Germany from which the samples containing pyridine were got, Dorn's or Pistorius' distillation apparatus is in use. In these the flame comes into direct contact with the boiler containing the alcohol, and any insoluble matter contained originally in the mass would probably be carbonised, yielding pyridine as a decomposition product. Recently the steam distillation apparatus has been more extensively in use in Germany than heretofore, and samples of amyl alcohol obtained in 1888 were found to be free from pyridine. The pyridine picrate forms needle-shaped crystals which are difficultly soluble in alcohol and in cold water. They melt at 144.5°, and are decomposed by strong acids or bases with separation of pyridine. Heated gently they are partially decomposed; strongly heated they explode. Their formula is $C_5H_5N \cdot C_6H_3(NO_2)_3OH$. To employ this picrate as a test for the presence of pyridine in amyl alcohol, picric acid is dissolved in the sample, the solution filtered and the crystals which separate on cooling recrystallised from hot water.—C. A. K.

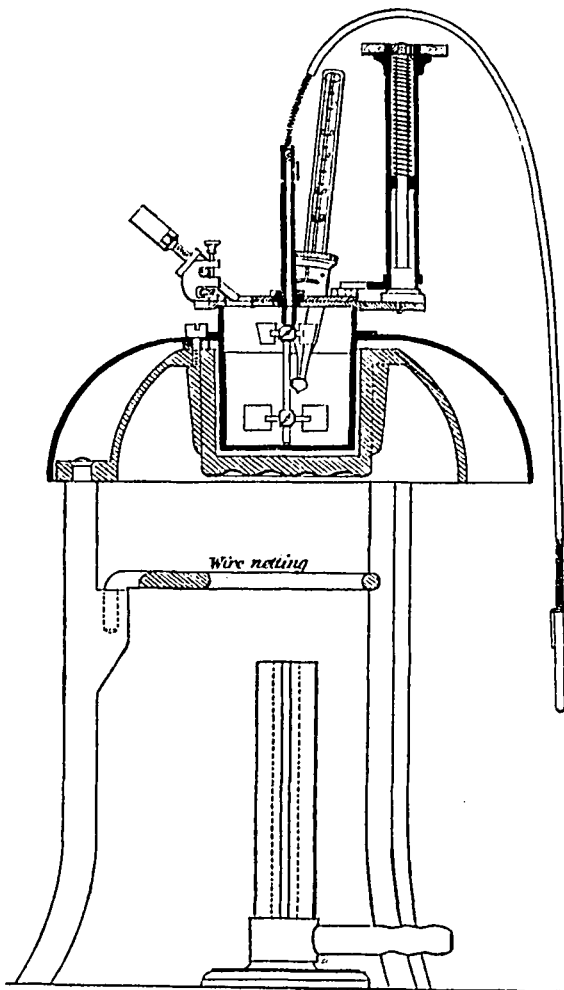
Distinction of Resoreinol from Carbolic Acid and from Salicylic Acid. H. Boddé. *Nederl. Tijdschr. v. Pharm. Chem. en. Toxikol.* 1889, **1**, 163.

A SOLUTION of resoreinol, either in water or in alcohol, gives a violet colouration on addition of a drop of sodium hypochlorite which soon turns yellow, and which on warming or on further addition of hypochlorite becomes dark yellow, red, or dark brown. One part of resoreinol in 10,000 parts of water can be detected by the violet colouration. Carbolic acid, salicylic acid, benzoic acid, and allied bodies do not give the violet colouration, the solution either remaining colourless or showing a slight fluorescence; on warming it turns slightly yellow. A solution of resoreinol treated with a few drops of ammonia and then with a few drops of sodium hypochlorite gives a red-violet colouration which turns yellow on standing and dark green on boiling. Salicylic acid, benzoic acid, and antifebrin give no colouration with this reagent; phenol gives a blue-green colouration.—C. A. K.

The Flashing Point of Mineral Lubricating Oil. Holde. *Mith. Königl. techn. Versuchs.* 1889, 64—75.

THE objections that are now conceded with respect to the methods of determining the flashing point of kerosene, formerly in use, are valid in the case of mineral lubricating oils. An apparatus on the lines of the Abel instrument is what is needed. A modification of the Pensky apparatus (Schädler's *Fette u. Oele d. Fossilien*, p. 443) was devised and is shown in the accompanying figure. The chief point in which it differs from the Abel oil-tester is the presence of an agitator with two sets of blades, so that both the oil and the atmosphere in the cup immediately above it are kept homogeneous. By this means local inequalities in the heating are obviated, a result which is also favoured by the even distribution of the hot gases from the lamp shown beneath the vessel itself. On this account there is little fear of arriving at an erroneous result by moderately rapid heating, a risk attending the use of the older forms. A rise of 12° C. per minute towards the close of the experiment may safely be adopted.

Some oils contain much water and give very irregular values for their flashing points. The presence of moisture may be recognised by heating the oil in a test tube in a clear oil-bath kept at about 140° C. As its temperature approaches 120° C. it begins to froth and may even "bump," while the film of it on the walls of the test tube is seen to be an emulsion of water and oil, and does not become clear even at 150° C. It is easy to distinguish these indications of the presence of water from those of entangled or dissolved air which is sometimes present. It is of course necessary in making this experiment (as in sampling the oil) to avoid even a trace of moisture in the vessels used.

Section through the Middle.

The results of numerous experiments demonstrating the closeness of the agreement of the figures obtained by the use of this apparatus, and the unreliability of more primitive methods, are recorded in voluminous tables.—B. B.

Detection of Mineral Oil in Fatty Oils. Holde. Mitth. Königl. techn. Versuchs. 1889, 75.

BENEDIKT's process ("Analysen der Fette und Wachsarten," p. 102), which consists in saponifying the oil with alcoholic potash, and adding ammonia to the solution, thus producing a turbidity if mineral oil be present, needs attention to be paid to his prescription, as a too weak alcohol fails to yield a clear solution, even with pure fatty oils; a large excess of ammonia, too, causes in any case a precipitate, the potash soap being thrown down.

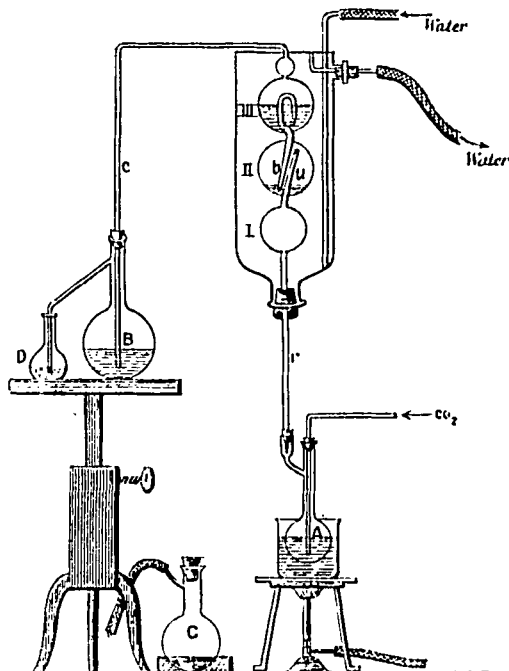
The author describes and recommends the following method:—A piece of caustic potash the size of a pea is heated in a test tube with about 5 cc. of absolute alcohol until dissolved; three or four drops of the suspected oil are then added, the heating continued for one minute and 3 or 4 cc. of distilled water added. If the solution remain clear a fatty oil only is present. Any little flocks of soap visible before the addition of water dissolve afterwards; even 1 per cent. of mineral oil causes the formation of a distinct turbidity.—B. B.

Estimation of Lime in Soils. F. Reverdin and C. de la Harpe. Chem. Zeit. 13, 726.

The lime present in soils serves different purposes. Not only does it act as a binding material for the soil and a promoter of the oxidation of the nitrogenous organic matters, but it also serves as an important plant food. And in these respects all lime salts do not act alike, for whilst, for instance, finely-divided calcium carbonate assists the formation of nitrates, calcium sulphate acts indifferently. It is therefore of importance to estimate the directly active lime in making an analysis of soil. P. de Mondesir does this by measuring the volume of carbonic acid gas which is liberated by treating the soil with tartaric acid. But this treatment causes the lumps of calcium carbonate to be dissolved, and as only the surface of the same is active, the method is unsatisfactory. The authors propose the following method: The sample of soil is digested on the water-bath for several hours with an excess of a mixture of equal molecules of sodium oxalate and carbonate. The sodium oxalate solution must have been standardised and the mixture be so concentrated that about 200 cc. will suffice for 20 grms. of the soil. The solution is filtered, the residue washed, the washings being added to the filtrate, and then the filtrate is acidified with hydrochloric acid. In most cases a precipitate of organic acids is produced, which must be filtered off. Sodium acetate is then added to the filtrate, and, after boiling, calcium chloride solution is added to precipitate the excess of oxalic acid, which is estimated with permanganate in the usual way. From the amount of oxalic acid which is found to be absorbed by the soil, may be calculated the quantity of active lime present. The carbonate of soda causes the magnesia to remain in the form of magnesium carbonate, whilst the sodium oxalate changes the finely divided calcium carbonate and other lime salts into calcium oxalate, but attacks only the surface of the lumps of calcium carbonate.—E. B.

Quantitative Determination of Methoxyl ($\text{O} \cdot \text{CH}_3$). R. Benedikt and H. Grüssner. Chem. Zeit. 13, 872—873.

THE method is a modification of that proposed by Zeisel (see this Journal, 1886, 335).

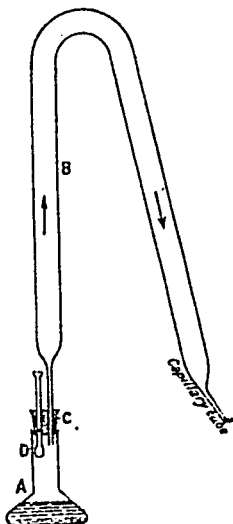


The substance (0.2 to 0.3 gm.) is placed in the flask A, the capacity of which is 30—35 cc., and there treated with 10 cc. of hydriodic acid of sp. gr. 1.70. A current of carbon

dioxide is passed through the flask, the heating of the latter being effected by a glycerol bath. The methyl iodide formed passes through the three bulbs I., II., and III. I. is empty, II. contains water, and III. water containing red phosphorus in suspension. These bulbs are surrounded by water, the temperature of which can be kept at 70° C. by passing in hot water; this is necessary in the determination of ethoxyl ($\text{O.C}_2\text{H}_5$). After passing the bulbs, the methyl iodide, which is quite free from hydriodic acid and iodine, is absorbed in the flask B by 5 cc. of a 40 per cent. solution of silver nitrate and 50 cc. of 95 per cent. alcohol. D is a safety flask containing 1 cc. of silver solution and 10 cc. of alcohol, but it was never found to contain a weighable quantity of silver iodide. The alcoholic solution is diluted with water, acidified with nitric acid, and the silver iodide filtered off and weighed.—C. A. K.

Volumetric Determination of Carbon Dioxide. F. Fuchs. Chem. Zeit. 13, 873.

The flask A is partially filled with dilute hydrochloric acid and this then saturated with carbon dioxide either by passing



the gas into the acid or by adding sodium carbonate. In this way the space above the acid becomes entirely filled with carbon dioxide at the end of the determination, thus avoiding any error due to the varying composition of the gaseous layer above the acid. The exit tube B is filled with dry air and inserted in the stopper C. The substance is placed in the small bottle D and held in a hole in the stopper, from which it is liberated by pressing down the glass rod above it. The liberated gas is measured as usual. Results varying from 0.07 to 0.37 per cent. from the theoretical value are given.—C. A. K.

Simultaneous Determination of Hydrogen and of Nitrogen. C. Gehrenbeck. Ber. 22, 1694-1696.

The combustion is carried out in an ordinary combustion tube open at both ends, in which the substance, intimately mixed with cupric oxide or with lead chromate, is placed. The stopper at the one end of the tube is furnished with a tube provided with a two-way cock, one passage of which communicates with an ordinary drying apparatus for air and oxygen, the other with a carbon dioxide generator. The calcium chloride tube is connected to the other end of the combustion tube, and attached to it is the apparatus for collecting the nitrogen (Schiff's apparatus was used). A second small calcium chloride tube is interposed between the two to prevent any sucking back of the liquid from the nitrogen apparatus into the calcium chloride tube. To conduct the analysis the whole apparatus is first filled with carbon dioxide (dried by means of sulphuric acid) and the

nitrogen determination made in the usual way. The two-way cock is then turned and the combustion completed in oxygen, this last being finally displaced by air.

Several test analyses are given with satisfactory results.

—C. A. K.

A New Method for the Quantitative Determination of Nicotine. M. Popovici. Zeits. f. Physiol. Chem. 13, 445.

Fifty grms. of the tobacco powder are moistened with 10 cc. of alcoholic soda solution (6 grms. of caustic soda in 40 cc. of water, with 60 cc. of 95 per cent. alcohol) and then extracted in a Soxhlet apparatus for 3-4 hours with ether. The extract is shaken with a moderately concentrated solution of nitrophosphomolybdic acid. The precipitate, which readily settles, after drawing off the ethereal layer is made up with water to 50 cc. and the nicotine set free by the addition of 8 grms. of finely-powdered barium hydroxide. The alkaline product containing free nicotine is filtered, and the rotary power of the filtrate determined in the polariscope; from this the quantity of nicotine can be found in a table prepared by the author, who employed ethereal solutions containing known amounts of nicotine, which was precipitated and treated as above, the rotary power being determined through a layer of the solution 20 cm. thick. Higher results are obtained by this process than by Kissling's; this is attributed by the author to the fact that some of the nicotine is lost in the latter process in distilling off the ether.—O. H.

New Books.

OEL- UND BUCHDRUCKFARBEN. Praktisches Handbuch für Firniß- und Farbenfabrikanten, von LOUIS EDGAR ANDÈS, Lack- und Firniß-Fabrikant. Mit 56 Abbildungen. All rights reserved. Wien, Pest und Leipzig. A. Hartleben's Verlag, 1889. London: H. Grevel and Co., 33, King Street, Covent Garden.

SMALL 8vo volume, bound in paper cover, price 4s. It contains 261 pages of subject-matter and 56 wood engravings. The treatment of the entire subject may be best exemplified by a summary of the contents, as follows:—Linseed oil; Purification and Bleaching of Linseed oil; Machinery and Apparatus used in purifying and bleaching oils. Chemical purification of Linseed oil. Oxidising agents for the conversion of Linseed oil into varnish. Theory of Varnish making. Manufacture of Linseed oil varnishes. The Colouring materials for paint—Artists' Oil—and Bright Printing Colours. The Colouring materials for Printers' black inks. Substitutes for Lampblack. Apparatus for Grinding and Triturating Colours. Mechanical arrangements for mixing the Binding materials with the Colours. Machines for grinding Colours. The Manufacture of Colours for Oil paints. Artists' Oil Colours. The Manufacture of Book Printing Colours. Composition Varnishes. Mixing the lampblack with the Varnish. Recipes for Printers' blacks. Improvements in the preparation of Book-printing Colours by Dr. H. Brackenbusch, &c., &c. The work closes with a full alphabetical index.

CHEMISCHE HOMOLOGIE UND ISOMERIE in ihrem Einflusse auf Erfindungen aus dem Gebiete der organischen Chemie. Eine patentrechtliche Studie vom Standpunkte des Chemikers. Von Dr. Otto N. Wirtz. 1889. Berlin: Verlag von Rudolf Mückenberger. London: H. Grevel and Co., 33, King Street, Covent Garden.

The object of this work (price 5s.) is briefly to discuss the relative positions of the organic chemist, discoverer, inventor, and patentee. It is an octavo volume of pamphlet form, containing 96 pages of text, and dedicated to Dr. H. Caro, the

well-known chemist, and Director of the Badische Anilin und Sodafabrik. It is subdivided as follows:—PART I. The Patent Laws of different Countries, and their Influence as regards Chemical Discoveries. PART II. Chemical Discovery and its Relations to Chemical Construction. PART III. Chemical Homology and Isomerism. PART IV. Homology and Isomerism as regards their Influence on the Patent System. PART V. Examples:—No. 1. Example of a Process which is distinguished from an already known Method by the Employment and Production of Homologous Substances. Consequences of this Homology. No. 2. Rejection of a Patent on the Ground of the Isomerism of the Ingredients used with Substances already known. No. 3. Granting of a Patent in spite of the Isomerism of the Products employed and produced with those of a Process already patented. No. 4. Granting of a Patent on the ground of the different Colour of the Isomerides obtained by the Process for which Protection is sought from that of already patented Isomerides. No. 5. Decision of the Government Jurisdiction of the 20th March 1889. PART VI. Final Conclusions.

CHEMISCH-TECHNISCHES REPERTORIUM. Uebersichtlich geordnete Mittheilungen der neuesten Erfindungen, Fortschritte und Verbesserungen auf dem Gebiete der technischen und Industriellen Chemie mit Hinweis auf Maschinen, Apparate und Literatur. Herausgegeben von DR. EMIL JACONSEN. 1888. *Zweites Halbjahr.—Erste Hälfte.* Berlin: R. Gaertner's Verlagsbuchhandlung. Hermann Heyfelder. London: H. Grevel and Co., 33, King Street, Covent Garden.

OCTAVO volume in paper cover; the first issue for the second half-year of 1888. It contains 160 pages of subject-matter, plentifully illustrated with wood engravings, and in the former the following subjects are treated of:—

Building materials, Cements, Artificial stone, Colouring Matters, Dyeing and Printing. Fats, Oils, Illuminating and Heating materials. Fermented liquors. Tanning, Leather, and Glue making. Textiles. Glass and Earthenware. Wood and Horn. India-rubber. Cements, Gums, &c. Lakes, Varnishes, and Paints. Metals.

Trade Report.

(From Board of Trade and other Journals.)

RAILWAY AND CANAL TRAFFIC ACT, 1888.

On page 249 of the *Board of Trade Journal* for September will be found the text of a memorandum issued by the Board of Trade on this subject on the 8th August last.

TARIFF CHANGES AND CUSTOMS REGULATIONS.

SWITZERLAND.

Classification of Articles in Customs Tariff.

Note.—Quintal = 220·4 lb. avoirdupois. Franc = 9 $\frac{1}{10}$ d.

The following decisions affecting the classification of articles in the Swiss Customs tariff were given by the Swiss Customs authorities during the month of July last:—

Fluoride of silicon and sodium (fluosilicate).—Category 17. Duty, 1 franc per quintal.

Paper used as blue for linen.—Category 38. Duty, 16 frs. per quintal.

Saccharin.—Category 246. Duty, 10 frs. per quintal.

Malt extract, in bottles or jars, with or without label.—Category 248. Duty, 10 frs. per quintal.

ITALY.

Classification of Articles in Customs Tariff.

Note.—Quintal = 220·4 lb. avoirdupois. Lire = 9 $\frac{1}{10}$ d.

The following decisions affecting the classification of articles in the Italian Customs tariff have recently been given by the Italian Customs authorities:—

Carbonate of lime obtained by precipitation.—Category 246a. Duty free.

Tanned hides, without hair, sewn.—Category 190d (5). Duty, 70 lire per quintal.

Pepsine acidulated with hydrochloric acid in the form of extract.—Category 62. Duty, 120 lire per quintal.

Non-alcoholic perfumeries, placed in little paper bags, are classified under "non-alcoholic perfumeries," the weight of the paper bags being included with the goods.

Vessels of celluloid for centrifugal machines intended for the bleaching of thread.—Category 226n. Duty, 11 lire per quintal.

UNITED STATES.

Recent Customs Decisions.

The following decisions affecting the classification of articles in the Customs tariff, and the application of the Customs laws of the United States, have recently been given by the United States Customs authorities:—

An article called "opium ashes," containing 6·02 per cent. of morphia, is dutiable at the rate of 10 dollars per pound under Schedule A. (T. I., 121).

Bauxite, ground or pulverised, is held to be free of duty under T. I., 604, on the ground of retaining its commercial designation irrespective of the condition in which it may be imported.

On the exportation of refined stearin, manufactured wholly from imported crude stearin, a drawback will be allowed of the duties paid upon a quantity of the imported material equal to the net weight of the exported article, less the legal retention of 10 per cent.

Bromo-fluoresceic acid, a derivative of coal tar, and itself a coal-tar colour, used in imparting colours to various fabrics, is classified under paragraph 82 (T. I., new), and dutiable at the rate of 35 per cent. *ad valorem*.

Ferro-silicon, ordinary pig-iron containing about 10 per cent. of silicon, and used in mixing with low grades of pig-iron to increase the percentage of silicon, and thereby strengthen the product, is dutiable as pig-iron at the rate of three-tenths of a cent per pound.

Fruit-juices, when containing over 20 per cent. of alcohol, should be classified under Schedule H. (T. I., 312), as "compounds or preparations of which distilled spirits are a component part of chief value," and subjected to a duty of 2 dollars per gallon.

So-called "jasmine water" is to be classified as a medicinal water under T. I., 93, and pay duty at the rate of 25 per cent. *ad valorem*.

Certain iron oxide used as a colour or paint to be classified under T. I., 87, for "colours and paints," and dutiable at the rate of 25 per cent. *ad valorem*.

Sylvénite is free as a manure salt.

Indian red (earth paint) is dutiable as colours at the rate of 25 per cent. *ad valorem*.

Albuminised paper is dutiable at 25 per cent. as paper not otherwise provided for.

"Chlorophyl" is dutiable as a vegetable colour at the rate of 25 per cent. *ad valorem*.

So-called creolen and ichthyol, products obtained from the distillations of English coal, are dutiable at the rate of 25 per cent. *ad valorem* as chemical compounds not otherwise provided for under T. I., 92.

So-called "primulin," a colour derived from coal tar, is dutiable under T. I., 82, at the rate of 35 per cent. *ad valorem*.